Transitions in the Strongly Collective Behavior of Dislocations and Interfaces at the Onset of the Nanoscale Regime

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Sandia National Laboratories
Livermore, CA

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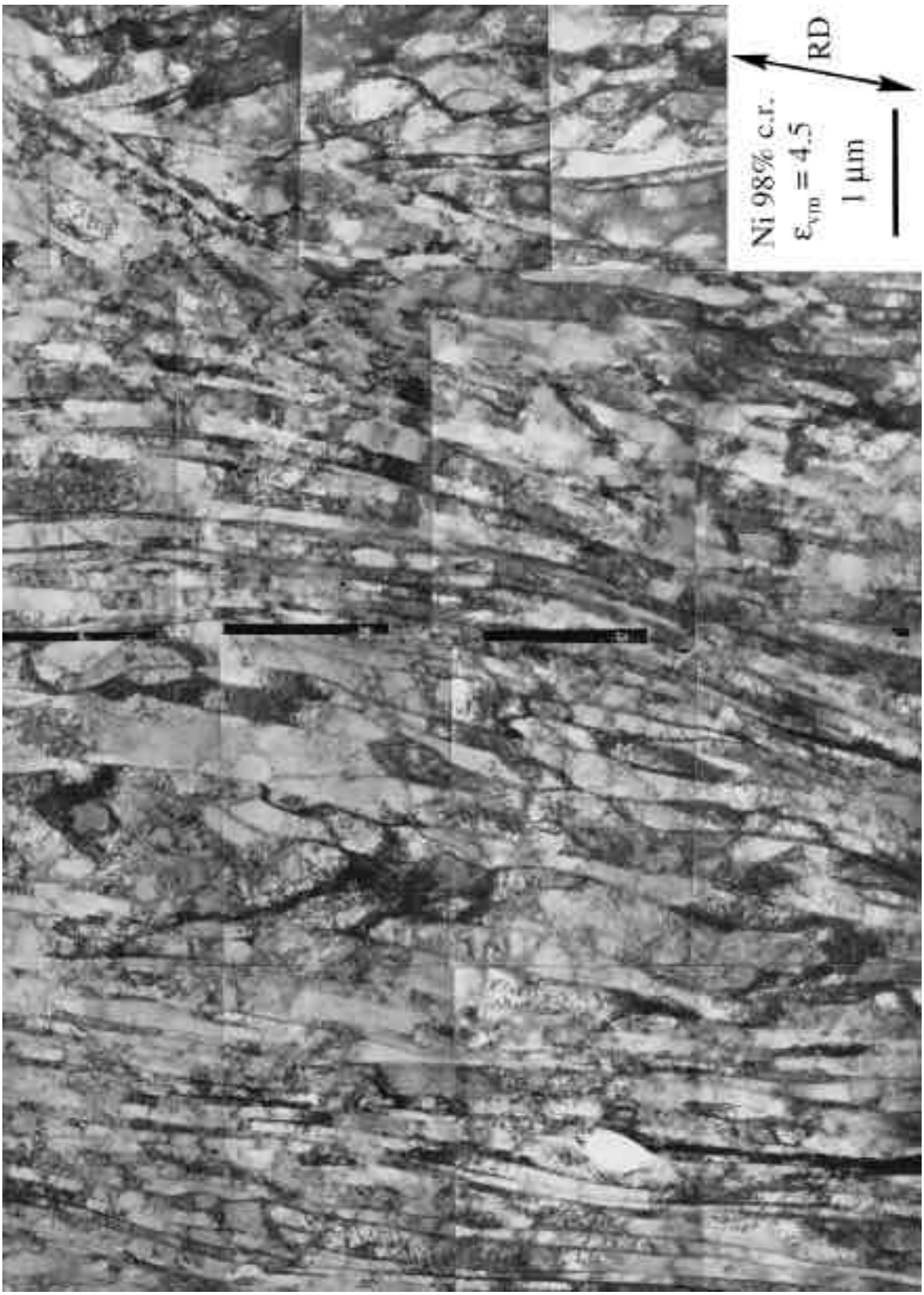
Sandia National Laboratories


Ni 98% c.r.
ε_{vm} = 4.5
1 μm
RD

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Executive Summary

The demand for fine scale components is dramatically increasing for a number of technologies that utilize micromechanical devices. To optimize properties including ductility and strength it has been proposed that many of those devices be made of metals and alloys whose microstructure scales from micrometers to nanometers. In response to these needs we are examining the mechanisms of local plastic deformation, in particular the nucleation, multiplication, mobility, interaction, and self organization of dislocations in fcc metals and alloys. We use microstructure evolution during deformation as a probe of fundamental properties. Quantitative analysis and measurements in the transmission electron microscope of structures that are created during monotonic deformation from small to large strains have shown that the structures maintain a similar character with increasing strain. Measurements include the averages and distributions of boundary misorientation angle/axis pair, inclination plane, spacing, and width for two types of deformation induced dislocation boundaries. Scaling of the distributions of these features and similitude provide governing principles for structure evolution. The development of these structures has been quantitatively linked to strength increases using an additive law for dislocation strengthening by low angle boundaries and a Hall-Petch type strengthening due to high angle boundaries.

Theoretical modeling of this evolution is therefore underway including the modeling of the spacing distributions and a multiscale coupling of three dimensional dislocation dynamics and continuum plasticity. The latter modeling effort examines the self-stress fields, stability and interactions between multi-Burgers vector dislocation boundaries to obtain information on boundary creation. Emphasis is placed on the boundary spacing both experimentally and theoretically since modeling the spacing evolution is the missing link. Success in this area would be a break through in understanding and modeling the mechanical behavior of metals.

The lower spatial limits of this behavior are being explored using graded nanostructures produced by sliding deformation. This new approach has shown a remarkable invariance on the pattern of boundary spacings with average sizes from 10,000 to 10 nm. This universal scaling behavior indicates a continuity in the processes that create and remove boundaries with increasing deformation. These processes are dislocation slip, short range dislocation interactions, and longer range interactions between boundaries. This size scale brings us into the range accessible by atomistic calculations. Exploration of the regime below 10nm is the next step.

Objectives

The demand for very fine scale components of complex shape, made from a wide range of materials from metals to semiconductors, is dramatically increasing for a number of technologies that utilize micromechanical devices. Important applications occur in the microelectronic, transportation, medical diagnostic, and implant industries. Fine scales pose new challenges in materials reliability and properties such as structural stability, mechanical strength, ductility, and resistance to fracture and fatigue. To optimize these properties, it has been proposed that many of these devices be constructed from submicrometer to nanoscale materials which often exhibit different properties from bulk materials. Desirable deviations from bulk behavior in these nanostructured materials include enhanced mechanical strength and ductility. The origins of these deviations have been postulated based on the expected restrictions on dislocation motion imposed by the small size scale.

Further insight into the mechanisms underlying these new properties can be obtained by investigating the transition(s) to nanoscale dimensions. We investigate such transitions within nanostructures obtained via plastic deformation. During deformation dislocations organize into patterns of dislocation rich boundaries enclosing dislocation poor regions. At very large strains, the spacing between dislocation boundaries reaches the nanometer scale. Our previous research (under our core BES funding for Science of Materials), part of which is included herein for this current review period, has shown that a number of microstructural parameters measured on such structures (e.g., dislocation boundary misorientation angle and spacing distributions) exhibit universal behavior during small to large deformations. Specifically, different materials and deformation conditions give rise to structures whose characteristics are remarkably similar apart from a change in scale. Integrated understanding of the origin of this behavior is just emerging.

We have shown breakdown in some aspects of this scaling behavior which occurs with increasing deformation of the submicrometer structures that have developed, indicating fundamental changes to the way high densities of dislocations and boundaries interact under an applied stress. In principle, additional breakdown in scaling may occur with further size reduction by deformation into the nanometer scale. While, the lower limits of this scale are yet unknown, we have recently demonstrated an incredible scale invariance of the spacing patterns from 10,000 to 10nm average sizes. We note in addition that this deformation process constitutes an unconventional route to produce nano-layered materials.

Our goals are to correlate the universal behavior mentioned above, and its potential breakdown, with changes in properties, microstructure, dislocation content, and boundary (interface) structure. Ideal materials are analyzed including single phase metals and eutectic alloys, e.g., nickel and copper-silver alloys. Fine-layered structures from ~1μm to ~10nm are produced in these materials by deformation under rolling, torsion (simple shear) and sliding friction. The layers created contain interfaces between rotated regions of the same metal, and interfaces that separate different metals. We are also starting to explore the stability of these structures at elevated temperatures. Perturbations in the scaling behavior of the structural elements are also used as probes for the mechanisms and microstructural changes accompanying the initial stages of the nucleation of recrystallization and grain growth.

High resolution transmission electron microscopy, automated diffraction analysis for crystal orientation and boundary misorientation, and fine electron probes for chemical analyses are used to characterize these interfaces, the dislocation structures between them, and the emergence of new plastic behavior at small scales. We also continue to improve the capabilities of our TEM analysis. We are particularly interested in the changes to the dislocation structures formed between the layers as the nanoscale regime is approached and scaling features may start

to breakdown. Conceptual models of plastic behavior will be developed based on experimental observations and appropriate simulation analyses.

These combined experimental and theoretical studies are intended to guide the development of new layered materials as well as robust constitutive models of deformation behavior to use in materials processing and applications from large scale forgings in industry to small scale devices.

Approach

Fine scale laminates, similar to the structures shown in Fig. 1, can be fabricated by different processes including a variety of deformation modes. It has been postulated that such laminates behave very differently than the same materials in bulk form. More explicitly, a modified relationship arises between strength, ductility, and size scale. In particular, increased strength coupled with enhanced ductility is possible. This anomalous behavior suggests fundamental changes to the range and nature of collective dislocation processes that operate during deformation.

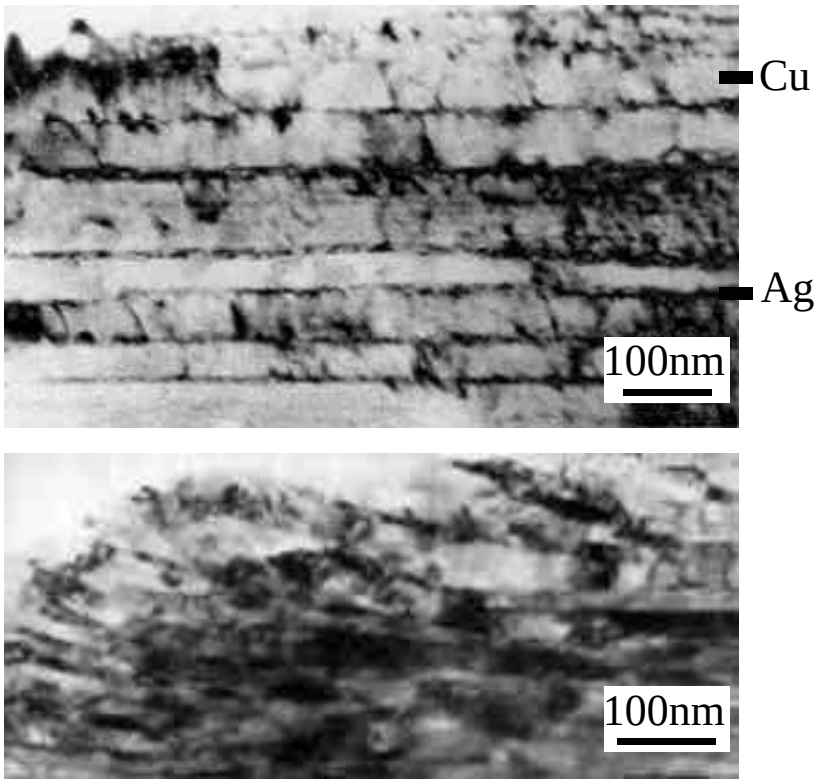

Fig. 1. a) Cu-Ag nano-laminate formed by the Taylor wire method (courtesy of J.D. Embury and H. Kung, LANL; Han *et al.*, 1998). As an analog to the bimetallic interfaces in (a), in b) a structure of ~25nm wide "monometallic" layers formed in copper by sliding over a steel platen under a large load. About 2% iron was introduced into the high purity copper during the sliding process. Dislocations are observed at high magnifications in between the deformation induced interfaces (Hughes *et al.*, 1995).

To develop a physical understanding of the crossover in behavior we perform quantitative microstructural characterization of metallic laminates and design new conceptual models. We will then combine this understanding with these modeling tools to tailor new nanostructured materials using conventional systems.

In principle, this crossover from bulk to nanoscale-type behavior may involve a sequence of transitions accompanied by structural changes. The latter may be subtle and gradual. Figure 2 shows a series of microstructures following increasing levels of monotonic deformation. The main feature of this series is the change in size scale of the dislocation structures. To precisely quantify these changes, and identify the transitions, we have measured several parameters that characterize these structures using transmission electron microscopy (TEM) and advanced semi-automatic Kikuchi pattern analysis. Figures 3 and 4 show the distributions with increasing strain of the spacing between dislocation boundaries and the misorientation angle across them. On average, these parameters span a wide range of values depending on the deformation conditions and material. Fortunately, evolutionary regimes are found where the scaled distributions for these parameters are independent of deformation conditions and material. That is, the distributions are universal.

Breakdown in this scaling behavior can be used to monitor transitions in structure. Dislocations in these structures form two types of boundaries of different size scale and morphology. The nearly planar boundaries (Fig. 2) are geometrically necessary boundaries (GNBs). GNBs separate differently deforming regions and are thus the markers of the deformation pattern including slip processes and loading conditions. Consequently our work emphazises these boundaries. The GNBs sandwich blocks of cells with short boundaries labeled incidental dislocation boundaries (IDBs) in Fig. 2. Data for the GNBs, plotted in Fig. 3, show a clear transition in the shape of the misorientation angle distribution, from the monomodal form observed at low strain (Fig. 3a) to the bimodal form observed at large strain (Fig. 3b). The additional peak at high angles in Fig. 3b is related to the accumulation of rotations associated with the development of a preferred crystallographic texture during deformation. The distribution of spacings for the same GNBs remains “self similar” throughout, even though the average boundary spacing has shrunk to the nanoscale range (Fig. 3c and d).

Different behavior is measured for the IDBs. Both the misorientation angle and boundary spacing distributions continue to scale following both small and large strain deformations (Fig. 4). One natural limit to this behavior will occur when the spacing between GNBs is so small that IDBs cannot form between them. The three-dimensional schematic diagram in Fig. 5 illustrates different arrangements of IDBs for different GNB spacings following very large strain as in Fig. 2d. At very low spacing, IDBs settle into a bamboo-like network confined between the GNBs. This dislocation structure of low angle IDBs in between the narrowly spaced GNBs is in stark contrast to the dislocation-poor regions in between the interfaces of bimetallic nanolaminates (Fig. 1a).

In summary, our objective is to first, address experimentally and theoretically the reasons why the GNB spacing distribution is largely insensitive to processes underlying this transition, and to what extent one should we really expect a transition in the GNB spacing distribution. Hindering such a transition might be the fact that the evolution of the IDB and GNB structures is coupled (Fig. 5). A similar consideration applies to the stress dependence of their spacings. Emphasis is placed on the boundary spacing since modeling the spacing evolution is the missing link in this field. Success in this area would be a break through in understatnding and modeling

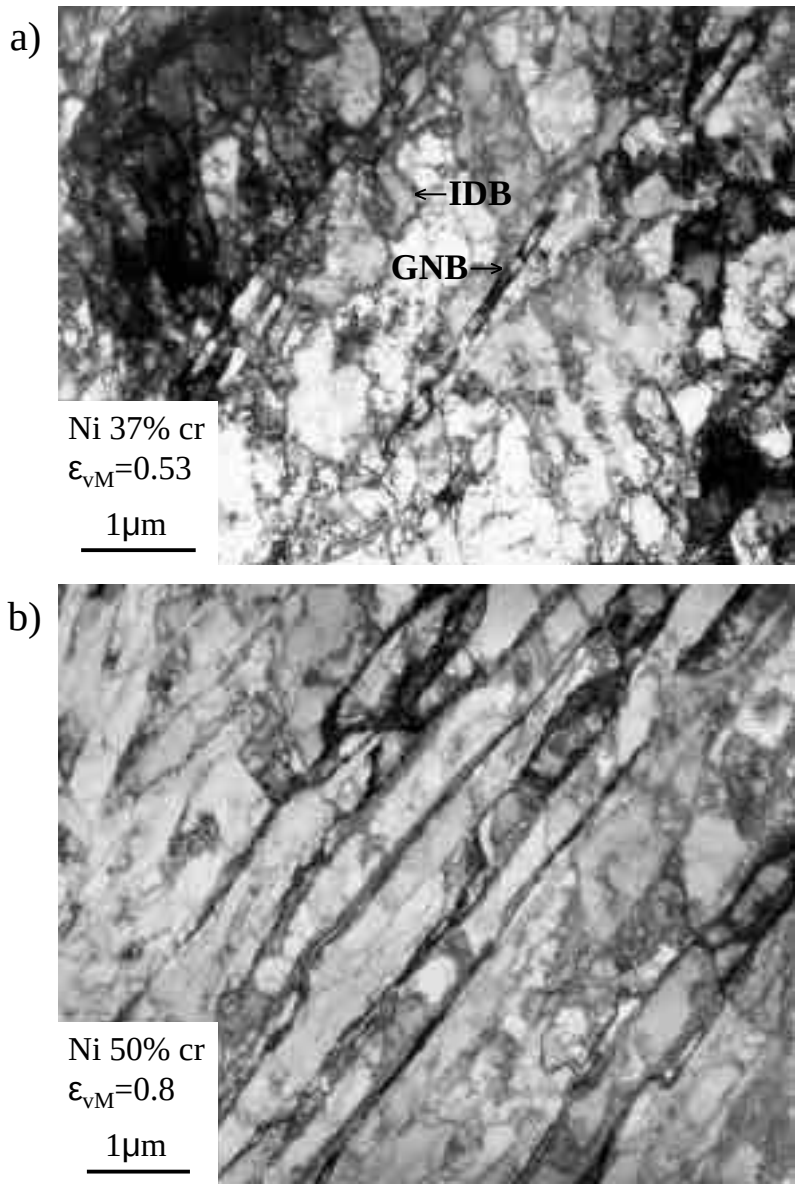

Fig. 2. TEM micrographs of high purity nickel (99.99%) following deformation by cold rolling at increasing levels of cold reduction (cr). The plane of view contains the normal direction and the rolling direction (RD). The RD in all frames is 11° clockwise of vertical. a) Following 37% cr, widely spaced GNBs enclose long blocks of equiaxed cells. The cell boundaries are marked IDBs. Measured average values of spacing and misorientation angle for the different boundaries are: D_{GNB}(av)=0.6μm. b) Following 50% cr, the structure remains qualitatively the same, but with finer boundary spacings and higher angles. Measured average parameters are: D_{GNB}(av)=0.39μm.

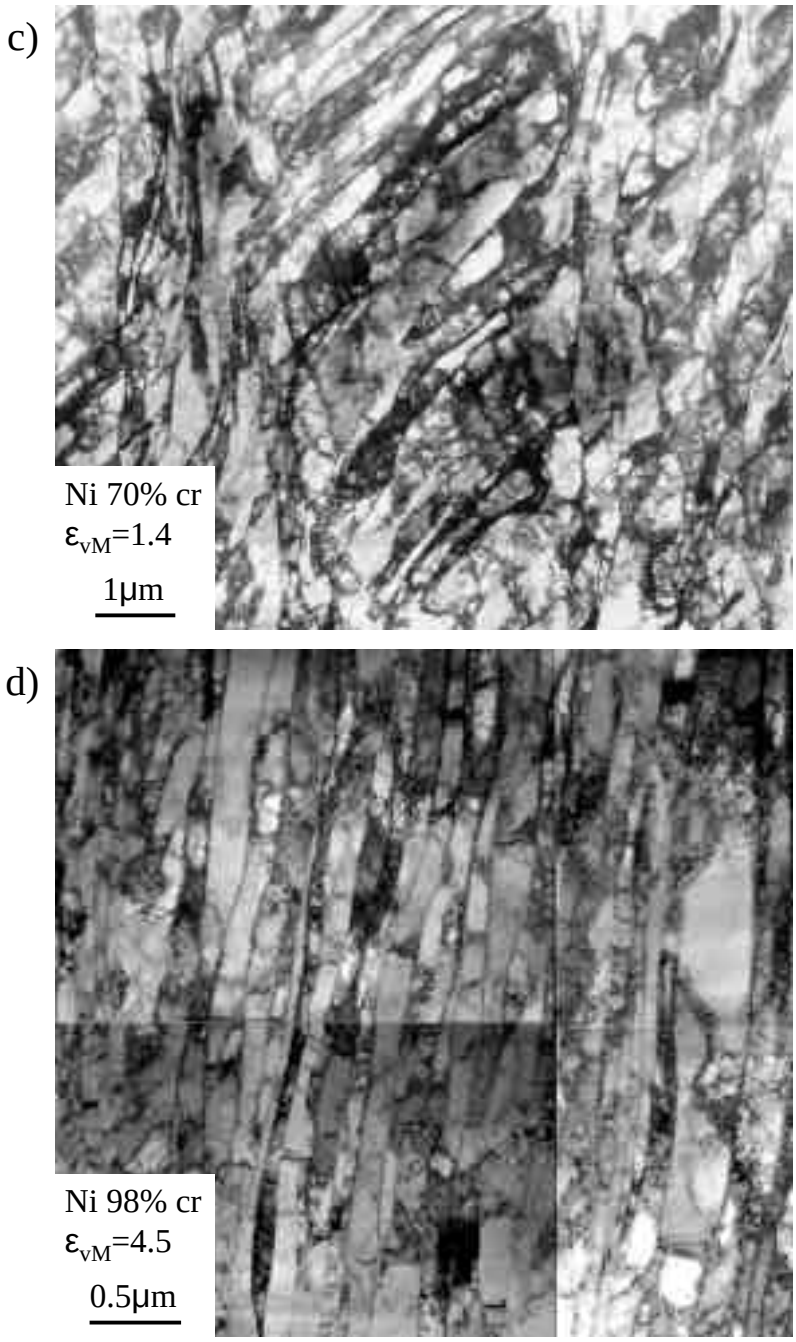

Fig. 2. (continued) c) An example of a transition structure following 70% cr, in which the inclination of some of the GNBs has become nearly parallel to the RD. Measured average parameters are: $D_{IDB}(av)$=0.42μm, $D_{GNB}(av)$= 0.28μm, $\theta_{GNB}(av)$=7.9°. b) Following 98% cr, typically a very fine layered structure is observed nearly aligned with the rolling plane. Measured average parameters are: $D_{IDB}(av)$=0.31μm, $D_{GNB}(av)$=0.13μm, $\theta_{IDB}(av)$=3.0°, $\theta_{GNB}(av)$=19.7°.

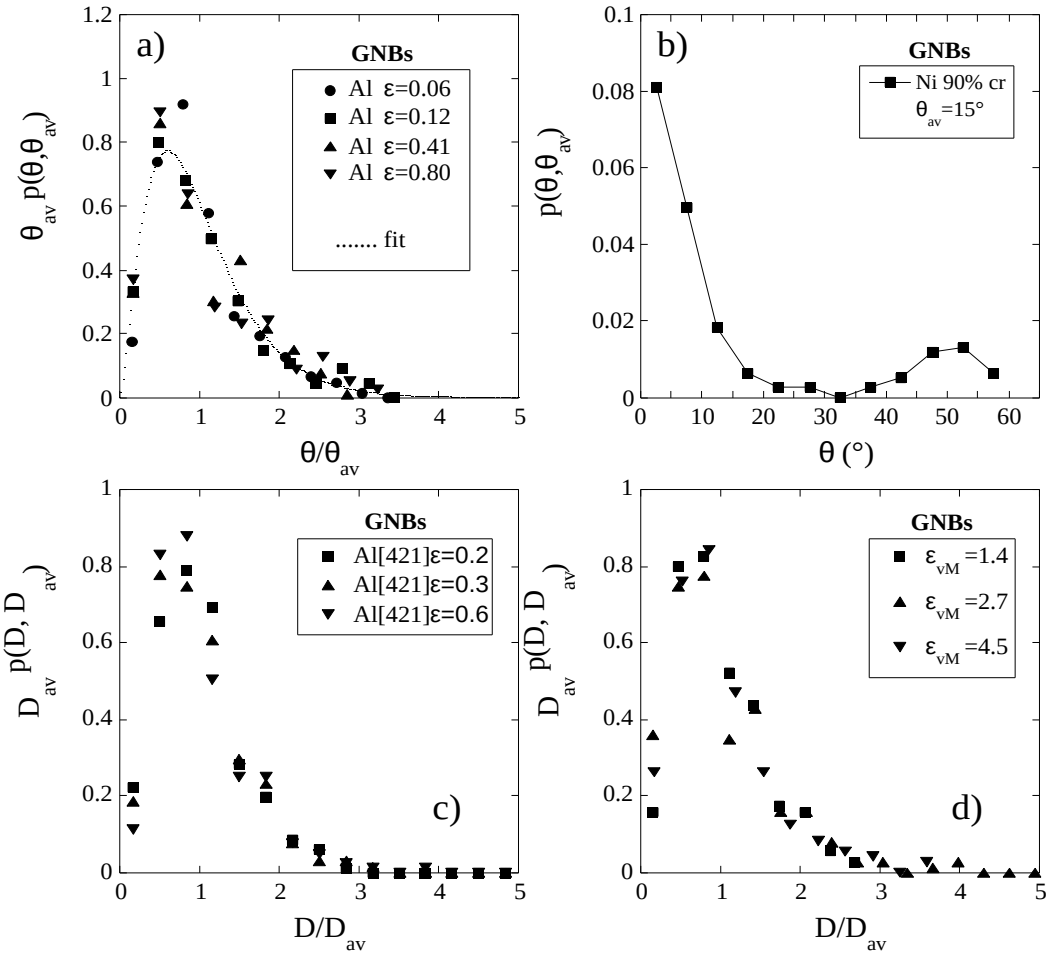

Fig. 3. a) Probability distributions for GNB *misorientation angles*, at small to medium strains, collapse into a single monomodal function when scaled by the average angle for cold rolled high purity aluminum. The average angle ranged from 1.2 to 7° (Hughes *et al.*, 1997). b) At large strain, a bimodal probability distribution of GNB *misorientation angles* is observed showing a breakdown of the low-strain scaling behavior (Hughes and Hansen, 1997). c) Probability distributions of GNB *spacings* at small to medium strains collapse into a single monomodal distribution when scaled by the average spacing which ranges from 4.5 to 1.3μm (Godfrey and Hughes, 2000). d) The same scaling relation for the GNB *spacings* in nickel is maintained to very large strains with very small spaciongs of 0.13μm, in contrast to the misorientation angle distributions.

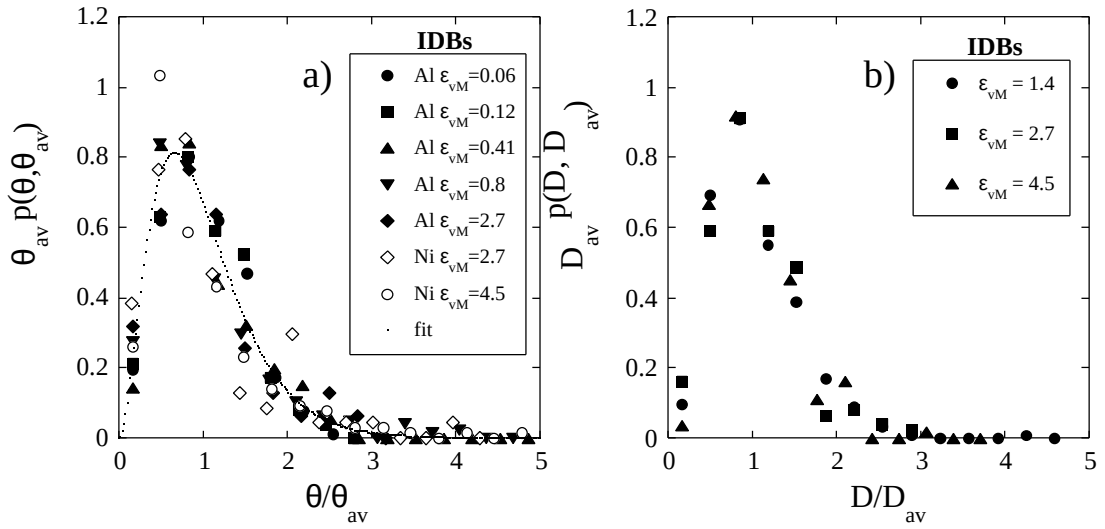

Fig. 4. a) Probability distributions for IDB *misorientation angles* at small to very large strains collapse into a single function when scaled by the average angle. The average angles range from 0.5 to 3.0° (Hughes *et al.*, 1998). b) Probability distributions for IDB *spacings* also collapse into a single function when scaled by the average spacing (see Fig. 2). Note that while the angle distribution is a full three-dimensional distribution, the distribution of spacings is two-dimensional due to the constraints of the measuring technique.

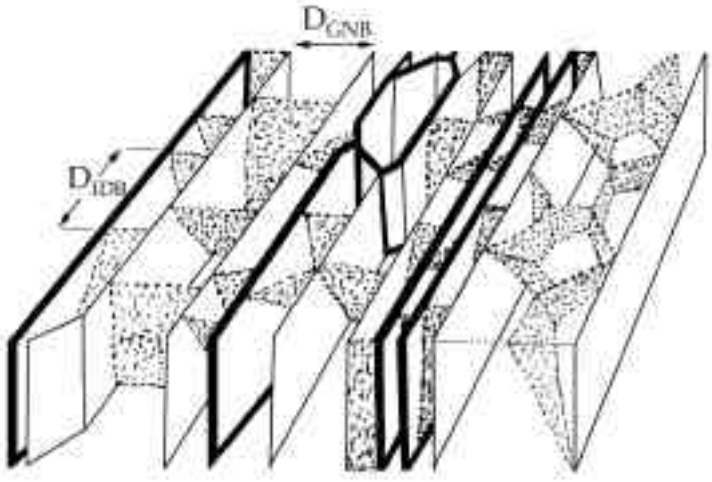

Fig. 5. Schematic drawing of a large strain layered structure (e.g., Fig. 2d) showing the bamboo-like network of IDBs with spacing D_{IDB}, in between the lamellar GNBs with nanometer scale spacing D_{GNB}. Note the difference in the IDB network for the widely spaced GNBs on the far right compared to the finer spaced GNBs on the left. In general, the lamellar GNBs end either at "h" junctions, like that at the bottom left corner, or at higher angle equiaxed subgrains, like that near the top center.

the mechanical behavior of metals. Third, we will probe for transitions that may occur at much larger strains. Structural comparisons with nanolaminates obtained via different processes will be exploited. Finally we will investigate the transition in angle distributions discussed above by examining and correlating changes in microstructure, crystallographic texture, and boundary dislocation content and arrangement at moderate strains. These combined investigations are aimed at developing a new understanding of deformation at small scales, and exploiting a potentially new class of deformation structures and processes using common materials.

Specific tasks we have undertaken include :

Large strain deformation tests on nickel samples using rolling (strains ~9), torsion (strains ~30), and sliding loads (strains>100).

Examine spatially graded structures created by sliding loads to pinpoint changes in dislocation mechanisms and structures in one sample.

Comparison with deformation structures and mechanisms in single phase nickel with those of eutectic alloy systems.

Modeling of the spacing distributions of dislocation boundaries and the dislocation structure within the boundaries.

Detailed studies of the first transition in the misorientation angle distribution. Nickel samples of different textures deformed to moderate strains in order to assess the role of a preferred texture evolution on the deformation mechanisms and structure. Manipulation of different textures developed during deformation using monotonic and cyclic deformation modes.

Facilities and Resources

Transmission electron microscopy is performed at Sandia National Laboratories on a state-of-the art 200kV field emission gun microscope equipped with electron dispersive spectroscopy (EDS), parallel electron energy loss spectroscopy (PEELS), scanning transmission electron microscope (STEM) mapping, and CCD and video cameras. The pole piece allows for large specimen tilts enabling diffraction contrast experiments. This TEM allows for nano-beam and convergent beam diffraction (CBED) as well as highly coherent 1nm fine probes for chemical analysis. Equally fine probes can also be used for CBED and the analysis of higher order Laue's zones for measurements of internal strain. The specimen goniometer is computer controlled. A semi-automatic technique for measuring crystal orientations with respect to the sample axis has been developed and installed. A CCD based system is being implemented for high accuracy orientation measurements. An in situ heating stage is also available. A 400kV dedicated high resolution TEM is available for atomic lattice imaging.

For larger scale analysis of structures, conventional and field emission gun scanning electron microscopes are available. We also have access to a SEM with an electron back-scattered diffraction pattern (EBSP) system for automatically measuring crystal orientations on a sample scale of millimeters. These SEM EBSP data will be used to supplement the TEM data.

Sample preparation and mechanical testing facilities exist at Sandia National Laboratories. The mechanical testing equipment includes several screw driven and servo-hydraulic systems with capabilities for uniaxial and multiaxial deformation conditions including torsion. Furnaces and environmental chambers are available for elevated and low temperature testing. Extensometers are available for strain measurement and control. Rolling mills and an alloy development and fabrication laboratory are available at Risø National Laboratories, through D. Hughes connection as a visiting scientist.

The theoretical work will be performed at Sandia National Laboratories and Washington State University which is equipped with a wide range of state-of-the-art single-processor workstations and clusters of processors from Sun, Silicon Graphics, Digital, and Intel.

Personnel

Darcy Hughes, Materials and Engineering Sciences Center, Sandia National Laboratories, CA: Principal Investigator: microscopy, mechanical properties and structure.

Mark Lyttle, Materials and Engineering Sciences Center, Sandia National Laboratories, CA post doctoral employee started 04/00: microscopy, mechanical properties; polycrystal modeling.

Andrew Godfrey, Materials and Engineering Sciences Center, Sandia National Laboratories, CA, post doctoral employee from FY97 through FY99, now at Tsinghua University Beijing China: microscopy and polycrystal modeling.

Maria Bartelt, Materials and Engineering Sciences Center, Sandia National Laboratories, CA limited term employee from FY97 through FY00, now at LLNL: Theoretical modeling.

John Korellis, Materials and Engineering Sciences Center, Sandia National Laboratories, CA: equipment designer for mechanical and friction experiments.

Hussein Zbib, Mechanical and Materials Engineering Department, Washington State University, Pullman, WA: dislocation dynamics and continuum modeling.

Shafique Khan, Mechanical and Materials Engineering Department, Washington State University, Pullman, WA, Ph.D. student started 01/00.

Sylvie Aubry, Materials and Engineering Sciences Center, Sandia National Laboratories, CA Staff just arrived 09/10/01. Theoretical modeling of microstructures.

Funding Summary

FY98 $100,000 (OBES Science of Materials SCW604)
FY99: $210,000 (includes $100,000 from OBES Science of Materials SCW604)
FY00: $430,000
FY01: $360,000

Collaborations and Relations to Other Programs:

The work on nanoscale layered materials is a new addition over the last two years, that fits within a long-term integrated effort at Sandia National Laboratories dedicated to correlating microstructural characteristics, and their evolution during plastic deformation and post-deformation annealing and processing, with measured materials properties and performance. Below, we highlight three of these programs, concerned with the fundamental nature of structure-controlled mechanisms of plastic deformation, recrystallization, and fracture.

This research has been facilitated by close interactions with other Office of Science programs and Laboratory Directed Research and Development (LDRD) initiatives, at Sandia and elsewhere. Close ties were made with the Metal Forming Group within the DOE Center of Excellence for the Synthesis and Processing of Advanced Materials. The Principal Investigator of this proposal (D.A. Hughes) was a task leader in that group. Her work has complemented the more applied activities of the group and its industrial partners. She has also maintained extensive informal collaborations with the metals deformation group at Risø National Laboratory in Denmark which now has a newly funded Center for Fundamental Research: “Metals in Four Dimensions”, Collaborators at Risø include Niels Hansen, Xiaoxu Huang, Wolfgang Pantleon and Niels Krieger Lassen. This collaboration includes both theoretical work on scaling and deformation mechanisms, microstructural analysis, as well as experimental exploration of new nanolaminate structures.

Nanocrystalline materials can undergo primary and secondary recrystallization at room temperature, often producing a structure of micron-sized grains in a finer-grained matrix. This coarsening process influences subsequent deformation behavior. In turn, details of recrystallization behavior, and resulting microstructure, depend sensitively on the starting structure, a feature often exploited in materials processing. A multi-level effort, supported by an LDRD and ASCII program at Sandia, is connecting dislocation, grain scale, polycrystal, and continuum modeling with experimental investigation of deformation and recrystallization. This work supports Sandia’s role in the forging of gas transfer components.

Sandia is also interested in developing diffraction contrast 3-D electron tomography in the transmission electron microscope for crystalline materials. Sandia is interested in developing this 3-D output for a number of defense and energy programs This output would an immediate input into structure codes and models of microstructure evolution, aging, and failure analysis for a wide range of applications from electronic devices, MEMS, and LIGA to metal forgings and bio materials. Collaboration through Sandia’s research foundations program, is being developed with Arlan Antolak, Analytical Materials Science Department, Sandia National Laboratories, CA who is active in the area of 3-dimensional reconstruction techniques for x-ray and ion beam tomography. This new capability would greatly enhance the TEM analysis of the nanostructures and dislocation boundaries of interest to our OBES program.

We also benefit from extraordinary local expertise in molecular dynamics techniques and finite-element and gridless methods, as well as newly developed Interfacial Force and Scanning

Tunneling Microscopy systems adapted to apply controllable stresses to microscopic samples in the other OBES programs at Sandia, CA.

Informal collaborations were made on texture and microstructure evolution with Hans Rudolf Wenk, Department of Geology, University of California, Berkeley, CA; Ricardo Lebensohn, Instituto de Fisica Rosario (CONICET-UNR) 2000 Rosario, Argentina; and Ashish Kumar, Formerly Division of Engineering Brown University, Providence R.I. now with McKinsey Associates.

Journal Publications

1. D. A. Hughes, and N. Hansen, “Graded Nanostructures Produced by Sliding and Exhibiting Universal Behavior,” *Phys. Rev Lett*., 87, issue 13 (2001) 135503.

   Physics Review Focus September 13, 2001 “Small Structures Act Big”

2. D. A. Hughes, "Deformation microstructures and selected examples of their recrystallization," Proceedings of the 21st Risø International Symposium: Recrystallization - Fundamental Aspects and Relations to Deformation Microstructures, ed. N. Hansen, et al. (Roskilde, Denmark: Risø National Laboratories, 2000), 49-70. *Journal of Surface and Interface Analysis*, 31, (2001) 560-570.

3. M. C. Bartelt, and D. A. Hughes, “Spatial Order and the Evolution of Deformation -Induced Structures,” *submitted*, (2001).

4. D. A. Hughes, S. M. A. Khan, A. Godfrey, and H. Zbib, Internal Structure of Deformation Induced Planar Dislocation Boundaries,” *Mater. Sci. Eng. A.* 309-310 (2001) 220-226.

5. D.A. Hughes, Microstructure Evolution, Slip Patterns and Flow Stress, in press *Mater. Sci. Eng.* March 2001.

6. D. A. Hughes and N. Hansen, “Microstructure and Strength of Nickel at Large Strains,” *Acta Mater.*, 48 (2000) 2985-3004.

7. Godfrey, and D. A. Hughes, "Scaling of Deformation Induced Dislocation Boundaries,” *Acta Mater.*, 48, (2000), 1897-1905.

8. N. Hansen, X. Huang and D. A. Hughes, “Microstructural Evolution and Hardening Parameters,” *Mater. Sci. and Eng.*, A317 (2001) 3-11.

9. D. A. Hughes, R. Lebensohn, H. R. Wenk, and A. Kumar, “Stacking Fault Energy and Microstructure Effects on Torsion Texture Evolution,” *Proc. Roy. Soc. London*, A 456 (2000), 921-953.

10. M. A. Miodownik, A. W. Godfrey, E. A. Holm, D. A. Hughes, “On Boundary Misorientation Distribution Functions and How to Incorporate Them into 3D Models of Microstructural Evolution,” *Acta Mater.*, 47, (1999), 2661.

11. D. A. Hughes, D. C. Chrzan, Q. Liu, and N. Hansen, “Scaling of Misorientation Angle Distributions,” *Phys. Rev. Lett.*, 81, (1998), 4664-4667.

Refereed Proceedings

12. D. A. Hughes, and N. Hansen, "Recrystallization in Subdivided Deformation Microstructures," Proceedings of the First Joint International Conference on Recrystallization and Grain Growth, ed. G. Gottstein and D. A. Molodov (Aachen: Springer Verlag, 2001) 659-668.

13. S. M. A. Khan, H. M. Zbib, and D. A. Hughes, "Stress Patterns of Deformation-Induced Planar Dislocation Boundaries," Material Instabilities and Patterning in Metals, ed. H. M. Zbib, et al. (Warrendale, PA: MRS, 2001) Volume 683E, BB.3.8.

14. M. T. Lyttle, and D. A. Hughes, "Nano-lamellar Structures in a Rolled Cu-Ag Alloy," Material Instabilities and Patterning in Metals, ed. H. M. Zbib, et al. (Warrendale, PA: MRS, 2001), MRS Proceedings Volume 683E, BB.1.4.1-6. .

15. D. A. Hughes and N. Hansen, “Characterization of Sub-Micrometer Structures in Heavily Deformed Metals Over the Entire Misorientation Angle Range,” Ultrafine Grained Materials, eds. R. S. Mishra, S. L. Semiatin, C. Suryanarayana and N. N. Thadhani (Warrendale: The Minerals Metals and Materials Society, 2000).
16. D. A. Hughes, “Distributions of Low and High Angle Boundaries in Deformed Metals,” *Recrystallization ’99, The Fourth International Conference on Recrystallization and Related Phenomena*, eds. T. Sakai and H. G. Suzuki (Sendai, Japan: JIMIS, 1999) p.111-118.
17. D. A. Hughes, and A. Godfrey, "Dislocation Structures Formed during Hot and Cold Working," *Hot Deformation of Aluminum Alloys II,* ed. T. R. Bieler, L. A. Lalli and S. R. MacEwen (Warrendale: The Minerals, Metals and Materials Society, 1998), 23-36.

Invited Lectures

keynote lecture:
D. A. Hughes, “Deformation Microstructures and Selected Examples of their Recrystallization,” 21st International Risø Symposium, *Recrystallization- Fundamental Aspects and Relations to Deformation Microstructure*, Roskilde, Denmark, September 2000.

plenary lecture:
D. A. Hughes, “Microstructure Evolution, Slip Patterns and Flow Stress,” International Conference on Strength of Metals and Alloys 12, Asilomar, CA, August 2000.

D. A. Hughes and N. Hansen, “Recrystallization in Subdivided Deformation Microstructures," Proceedings of the First Joint International Conference on Recrystallization and Grain Growth, Aachen Germany, August 2001.

D.A. Hughes, “Characteristics of Deformation-Induced Boundaries”, Computational Materials Science Network Workshop on Microstructural Effects on the Mechanics of Materials, Berkeley, June 2001

D. A. Hughes, “The Evolution of Deformation Microstructures and Grain Subdivision,“ MIT Cambridge MA, March 21 2001

D. A. Hughes, “The Evolution of Deformation Microstructures and Grain Subdivision,” Univ. Virginia Charlottesville VA, February , 2001

D. A. Hughes and A. Godfrey, “Recovery and Recrystallization within a Layered Deformation Microstructure,” Thermec 2000, Las Vegas, NV, December 2000.

D. A. Hughes, “Spatial Distributions of Dislocation Structures from Millimeters to Nanometers,” MRS symposium Multiscale Materials Modeling, Boston, MA, November 2000.

D. A. Hughes, “Grain Subdivision and Microstructure Evolution During Deformation,” Cornell University, Ithaca, NY, October 2000.

D. A. Hughes, S. M. A. Khan, A. Godfrey, and H. Zbib, Internal Structure of Deformation Induced Planar Dislocation Boundaries,” *Dislocations 2000*, Gaithersburg, MD, June 19-22, 2000.

D. A. Hughes, “Microstructures in Deformed Materials,” Workshop on Microstructure in Materials, Brown University, Providence, RI, December 1999.

D. A. Hughes, “Distributions of Low and High Angle Boundaries in Deformed Metals,” *Recrystallization ’99, The Fourth International Conference on Recrystallization and Related Phenomena*, Tsukuba City, Japan, July 1999

D. A. Hughes, “Grain Subdivision and Microstructure Evolution During Deformation,” Colloquium, Johns Hopkins University, May, 1999.

D. Hughes, “Grain Subdivision and Microstructure Evolution During Deformation,” Colloquium, University of Michigan, April, 1999

D. A. Hughes, “Dislocation Cell and Boundary Formation,” American Physical Society, Atlanta, March 1999.

D. A. Hughes, "Cold Deformation and the Nucleation Aspects of Recrystallization," *Plasticity '99 Constitutive Modeling of Inelastic Deformation and Phase Transformation,* Cancun, January 1999.

D. A. Hughes, and A. Godfrey, "Microstructure and Texture Development of Compressed [421] Single Crystals and [421]/[13 4 -3] Bicrystals," *Plasticity '99 Constitutive Modeling of Inelastic Deformation and Phase Transformation*, Cancun, January 1999.

D. A. Hughes, and A. Godfrey, "Dislocation Structures Formed during Hot and Cold Working," *Hot Deformation of Aluminum Alloys II*, TMS, Rosemont, October 1998.

Selected Professional Activities

Member Editorial Board: *International Journal of Plasticity*

Visiting Scientist Risø National Laboratory one month/year 1989 to present.

Organized special symposia for Plasticity ’99: “Modeling Plasticity at Different Length Scales” and “Recrystallization”

Organized MRS symposium “Material Instabilities and Patterning in Metals”, April 2001, San Francisco

Awards and Recognition

Physics Review Focus September 13, 2001 “Small Structures Act Big”

S. M. A. Khan, H. M. Zbib, and D. A. Hughes, "Stress Patterns of Deformation-Induced Planar Dislocation Boundaries," Material Instabilities and Patterning in Metals, ed. H. M. Zbib, et al. (Warrendale, PA: MRS, 2001): Best poster MRS San Francisco 2001.

Summary of Accomplishments

Our accomplishments cover the evolution of deformation micro and nanostructures, the role of deformation slip pattern and alloy compositon on dislocation boundary formation, the stability of this pattern to temperature increases and the development of experimental techniques to achieve these goals.

- Discovered that the probability distributions of dislocation boundary spacings remains the same when normalized by the average spacing for a wide strain range (ε_{vM}=0.2 to ε_{vM}=4.5), different materials and deformation conditions indicating that the same deformation mechanisms are operative for different fcc materials in contacts, forgings, rolling, torsion, compression (pub. 1, 2, 7, 15, 17).
- Established that the same scaling behavior of boundary spacings is observed from 10,000 to 10nm indicating that the same fundamental deformation processes of dislocation slip control the evolution over this range (pub. 1).
- Geometric and analytical models for the evolution of spacing probability distributions, that allow for either formation or coalescence of boundaries, were constructed providing good agreement with experiment (pubs. 3, 7).
- Developed stereological methods to measure spacing and probability distributions for the spacings of different types of deformation-induced dislocation boundaries imaged by TEM (pub. 7).
- Created novel technique to explore deformation behavior from 10,000 to 10nm with graded microstructures produced by sliding. ε_{vM}=100 (pub. 1)
- Observed that bimetallic interfaces in the Cu-Ag eutectic are efficient sinks for dislocations during deformation. Their constraint changes the number and pattern of dislocation boundaries that develop, thereby quickly creating nanolaminates with increasing deformation (pub. 14).
- Established the role of microstructure in crystallographic texture formation. Changes in stacking fault energy modify the different dislocation slip patterns within subdivided grains. New texture components are thereby created in a process that is at odds with the central Taylor theory (pub. 9).
- Simulated planar dislocation boundaries using a multi-scale coupling of a 3-D discrete dislocation dynamics code with the continuum finite element code. This method corrects for the boundary conditions of a representative volume element enabling us to study the stress fields and interactions between different boundary arrangements (pubs. 4, 13).
- Developed experiment to obtain deformed structures with a high density of recrystallization nuclei at the onset of dynamic recrystallization. TEM and scaling analysis make a direct links between deformed structure and the nucleation of recrystallization (pubs. 2, 12, 16).
- Predicted for the first time flow stress, from yield to large strains, using the measured microstructure parameters and a new model based on i) dislocation strengthening due to the presence of low angle boundaries and ii) grain boundary strengthening due to medium and high angle boundaries (pubs. 5,6,8)
- Captured changes in the hardening Stages III and IV quantitatively without invoking a change in mechanism (Pub. 6).

EVOLUTION OF DISLOCATION BOUNDARIES AND STRUCTURES

Andrew Godfrey and Darcy Hughes
Sandia National Laboratories, Livermore CA

Motivation:
All processing of materials' shapes, whether by rolling, forging, or milling involves plastic deformation. The deformation microstructures that develop control the materials properties, functionality and range of applications. Identification of microstructural relationships which are invariant and/or scale during straining are important towards obtaining both fundamental understanding of microstructural evolution and to construct predictive physical models of deformation.

Accomplishment:
An invariant form for the observed microstructural evolution has been found experimentally. A simple model has been proposed to explore the observed scale invariance. Single crystals of aluminum were compressed and polycrystals of nickel were rolled to different strain levels. Transmission electron microscopy was performed on the deformed samples to measure the dislocation structure as a function of material and evolutionary state. Distributions of dislocation boundary spacings and the misorientation angles across them were obtained. A scaling hypothesis, based on the average spacing, was applied to the widely varying distributions. Remarkably all of the scaled distributions collapse into a single distribution as shown in Fig. 6. This distribution is very similar to that obtained previously for the scaled distributions of boundary misorientation angle. Those scaled distributions remain invariant under a wide range of materials and monotonic processing conditions.

To investigate the origins of the scaled distribution a simple model that considers the effect of introducing new boundaries into a preexisting structure has been made. In this model new boundaries are introduced in between older boundaries based on simple rules for their location in a prexisting array of boundaries. The average spacing between boundaries is thus decreased in this scheme. The results of this model are shown in Fig. 7 in which the evolution of the scaled boundary spacings in the model is very similar to that observed experimentally in Fig. 6.

Significance: The observed universality reveals that the microstructure is related to processing by simple rules that can be incorporated into predictive models. These rules are valid for a wide range of materials and process conditions and are needed to direct model selection. Predictive models are essential to lower production costs and energy consumption in high volume, energy intensive metal forming industries.

Publications:
D.A. Hughes, D.C. Chrzan, Q. Liu, N. Hansen, "Scaling of Misorientation Angle Distributions," Phys. Rev. Let., 81, 1998, p 4664.

Godfrey, and D. A. Hughes, "Scaling of Deformation Induced Dislocation Boundaries," *Acta Mater.*, 48, (2000), 1897-1905.

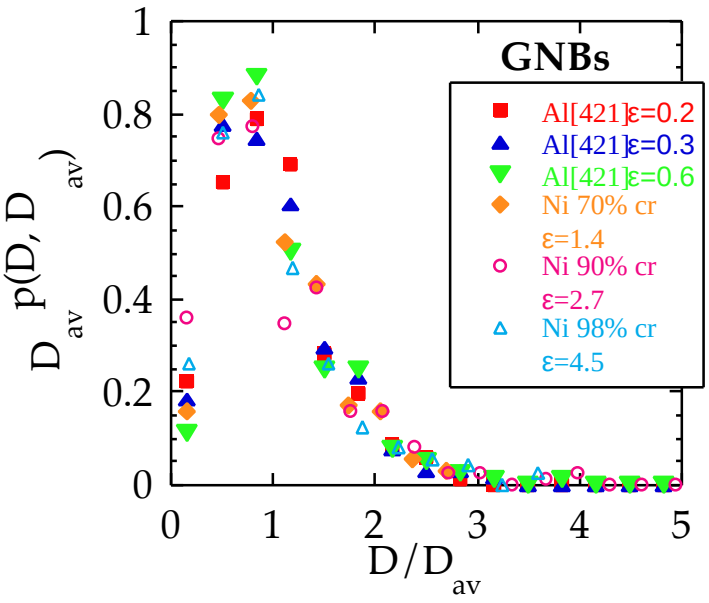

Fig. 6. Scaled probability distributions for GNB dislocation boundary spacings, D, for a wide strain range in aluminum and nickel. The average spacings, D_{av}, ranged form 4.5 to 0.13 μm.

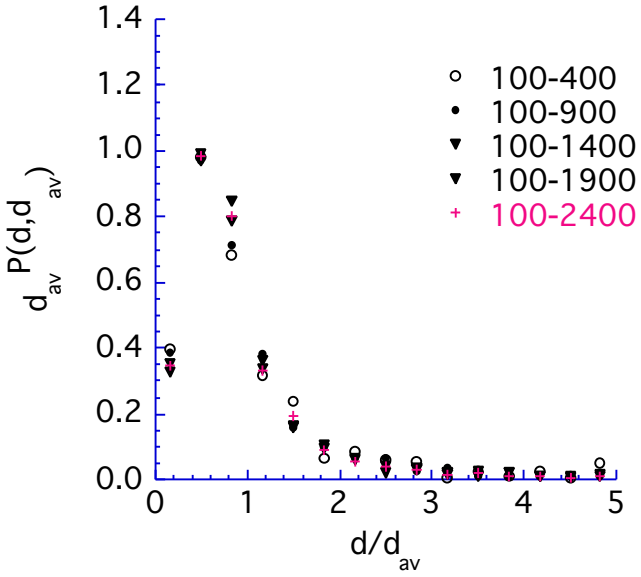

Fig. 7. Scaled spacing distributions obtained from a simple model that includes the effect of creating new dislocation boundaries in between the old ones. The observed shape of the caclulated distribution is similar to the experimental results. The number of new boundaries that has been added within the initial array of 100 boundaries is shown in the legend.

GRADED NANOSTRUCTURES PRODUCED BY SLIDING

[1]D.A. Hughes and N. Hansen[2]
[1]Sandia National Laboratories, Livermore CA, [2]Risø National Laboratory, Denmark

Motivation:
The demand for very fine scale components is dramatically increasing for a number of technologies that utilize micromechanical devices. To optimize properties, including ductility and strength, it has been proposed that many of those devices be constructed from metals and alloys whose microstructure scales from sub-micrometers to nanometers. Such metals are very difficult to produce, but cold deformation is a promising route. Large plastic deformation (strain) is required since even a one hundred times thickness-reduction in plate rolling of aluminum, copper or nickel can only bring the scale of the microstructure down to about 100-200 nm. A way to obtain extreme degrees of deformation is to press metal parts together and slide them against each other.
During deformation of metals microstructural universality has been shown for scales above a 150 nm as defined by the grain size or distance between boundaries. Probing this universality in the evolution at even finer nanometer scales is a second objective of the present analyses of friction samples.

Accomplishment:
Nanostructured copper was produced by deformation under large sliding loads. In the near surface layers, 10 nm scale microstructures form which coarsen with increasing depth from the surface as the degree of deformation diminishes Fig. 8 and 9. We use these graded structures because they present a unique opportunity to quantitatively characterise microstructures ranging continuously from 10 to 1000 nm in the same sample. We thereby explore a scale not accessible to conventional deformation experiments, in which the lower limit is about 100nm, or atomistic calculations, in which the upper limit is about 10nm. Our findings reveal the unanticipated result that universal scaling of deformation microstructures exists over the extraordinary size range from 10,000 to 10nm fig. 10. Our results push the limits of scale within an order of magnitude of the ultimate size limit at which we expect crystallinity to be destroyed.

Significance:
This universal scaling, coupled with the continuous reduction in average spacing with increasing deformation, indicates that there is a continuity of the processes which create new boundaries and those that remove them, from low to very high strain. These processes are dislocation slip, short range dislocation interactions and longer range interactions between the boundaries. The reduction in size with estimated strain shows a continuous refinement without saturation, demonstrating clearly that cold deformation processes have the potential to produce microstructures with characteristic spacing down to 10 nm following very high strains. An order of magnitude decrease in size is realized by adapting this friction process compared to other bulk deformation processes (e.g. rolling, equal channel angular pressing, drawing, and accumulative roll bonding).

Publication:
D. A. Hughes, and N. Hansen, "Graded Nanostructures Produced by Sliding and Exhibiting Universal Behavior," *Phys. Rev Lett..*, 87, issue 13 (2001) 135503.
Physics Review Focus September 13, 2001 "Small Structures Act Big"

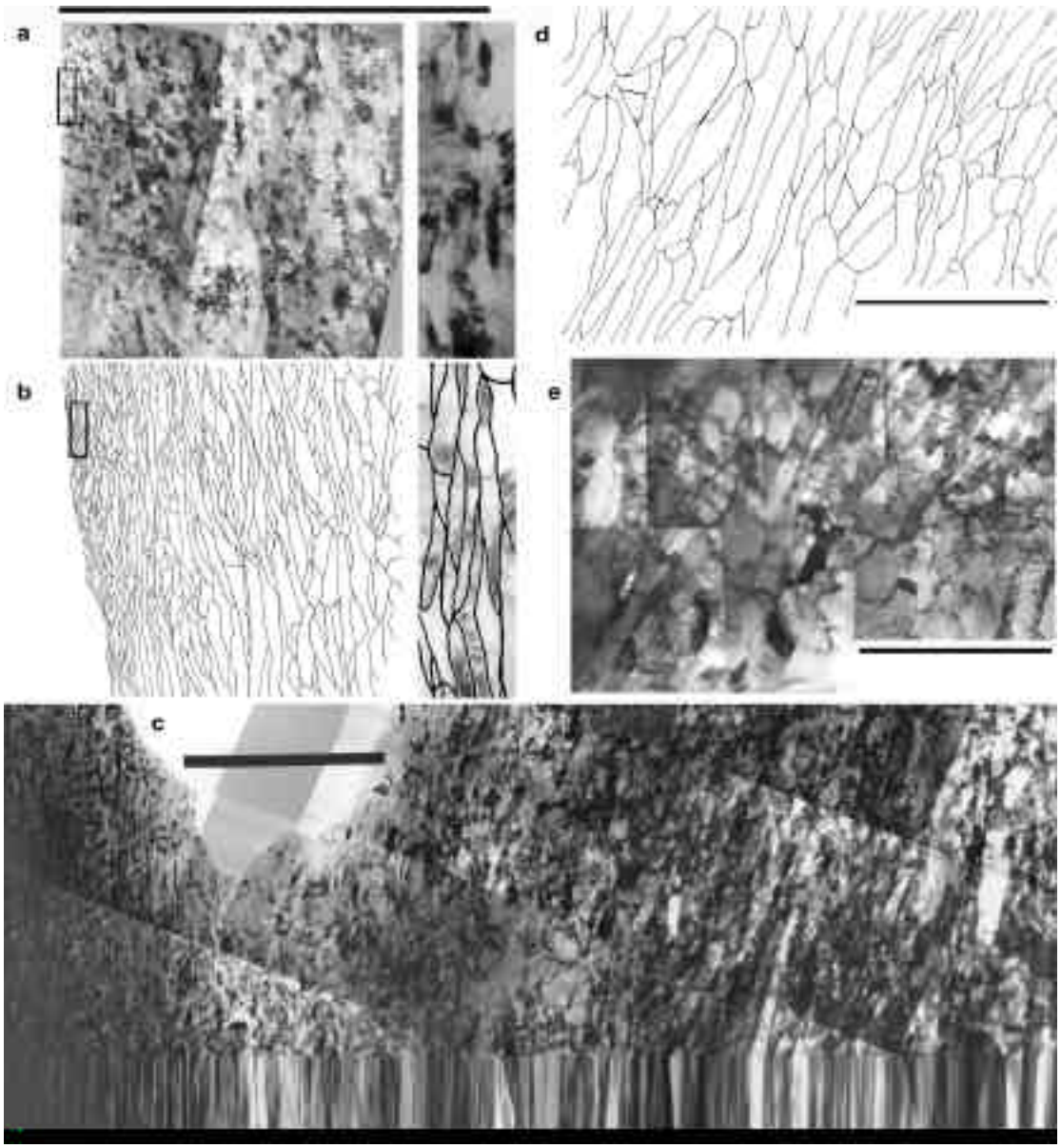

Fig. 8. Graded nanostructures produced by friction deformation as viewed in cross-section by TEM. a) micrograph and b) tracing of extended boundaries in copper following 127 mm of sliding under a 12 MPa applied normal pressure, the left side is coincident with the surface. Rectangualr boxes show location of adjacent high magnification excerpts illustrating a layer with 12nm spacing. These rectangles are 70nm wide. c) following 127 mm of sliding with 22 MPa normal pressure; the surface is coincident with the left side. (The pale gray region underneath the scale marker is the hole produced when making the electron transparent TEM foil.) d) tracing and micrograph e) of sample in a) but at 20 μm below the surface. Scale markers are 2 μm.

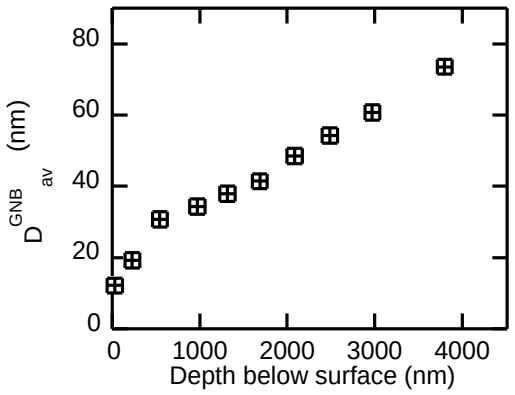

Fig. 9. Average perpendicular spacing between boundaries D_{av}^{GNB} vs. depth below the surface for a friction sample with 12 MPa normal pressure.

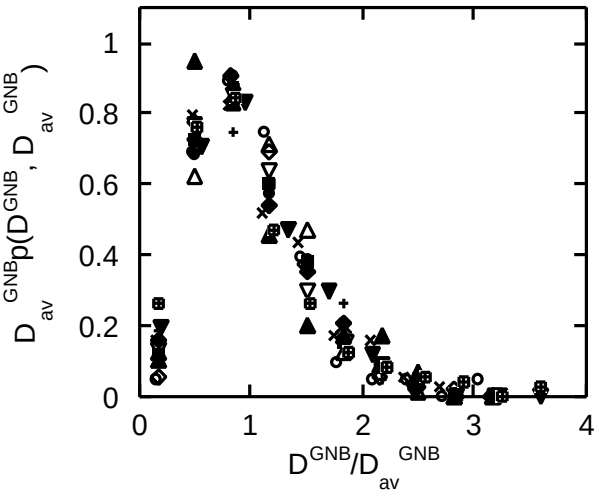

Fig. 10. Probability distributions of nanometer scale boundary spacings produced by friction as a function of depth normalized by their average spacing (see Fig. 2) compared to distributions at larger scales in rolled Ni. Friction: open circles: D_{av}^{GNB}=12 nm, open squares: D_{av}^{GNB}=19 nm, open diamonds: D_{av}^{GNB}=31 nm, open up triangles: D_{av}^{GNB}=35 nm, open down triangles: D_{av}^{GNB}=38 nm, solid circles: D_{av}^{GNB}=41 nm, solid squares: D_{av}^{GNB}=49 nm, solid diamonds: D_{av}^{GNB}=55 nm, solid up triangles: D_{av}^{GNB}=61 nm, solid down triangles: D_{av}^{GNB}=73 nm, squares with cross: D_{av}^{GNB}=171 nm; Rolling: plus: Ni 70% reduction, D_{av}^{GNB}=280 nm and cross: Ni 98% reduction, D_{av}^{GNB}=133 nm.).

DEFORMATION MICROSTRUCTURE EVOLUTION AND SIMILAR BEHAVIOR

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Motivation:
New tools for characterizing and analyzing microstructures have been developed in the last five years which create a firmer basis to explore the relationship between microstructure and the mechanisms that create them. A central question regards the identification of the different microstructural elements and their persistence from small to large strain deformation.

Accomplishment:
A quantitative microstructural analysis was performed on pure polycrystalline nickel (99.99%) cold rolled to reductions including 10, 20, 70, 90 and 98% (ε_{vM} 0.1 to 4.5). Applying transmission electron microscopy (TEM) techniques, key structural parameters, such as spacing between dislocation boundaries and high angle boundaries, as well as the misorientations across their boundaries, have been measured and analyzed. Application of scaling and similitude hypotheses to these microstructural parameters and their distributions revealed that the structures are homologous with increasing strain. More specifcally it is found that the separate spacing distributions of GNBs and IDBs remain the same with increasing strain when the distributions are normalized by their average values. The same result is found for the misorientation angle distribtions for IDBs. As further indication of the cooperative development of the structure it is observed that the average GNB width decreases in proportion to its spacing, Fig. 11. This similar behavior indicates that the measured parameters capture the important features of the structure. The following conclusions are reached. The key structural element is a cell block, composed of cell block boundaries (GNBs) and cell boundaries (IDBs) (*cf.* Fig. 3). For both types of boundaries, boundary spacing and width decreases and the misorientation angle increases with increasing strain, Fig. 12. Saturation of these parameters was not observed.

Significance:
Scaling and similitude provide governing principles for structure formation from small to large strain. The realization that certain aspects of monotonic deformation behavior is “universal”, independent of material and deformation conditions, raises the possibility that a usefull quantititative description of plastic deformation in terms of structural changes is feasible.

Publications:
D. A. Hughes and N. Hansen, “Microstructure and Strength of Nickel at Large Strains,” *Acta Mater.*, 48 (2000) 2985-3004.
D.A. Hughes, Microstructure Evolution, Slip Patterns and Flow Stress, in press *Mater. Sci. Eng.* March 2001.

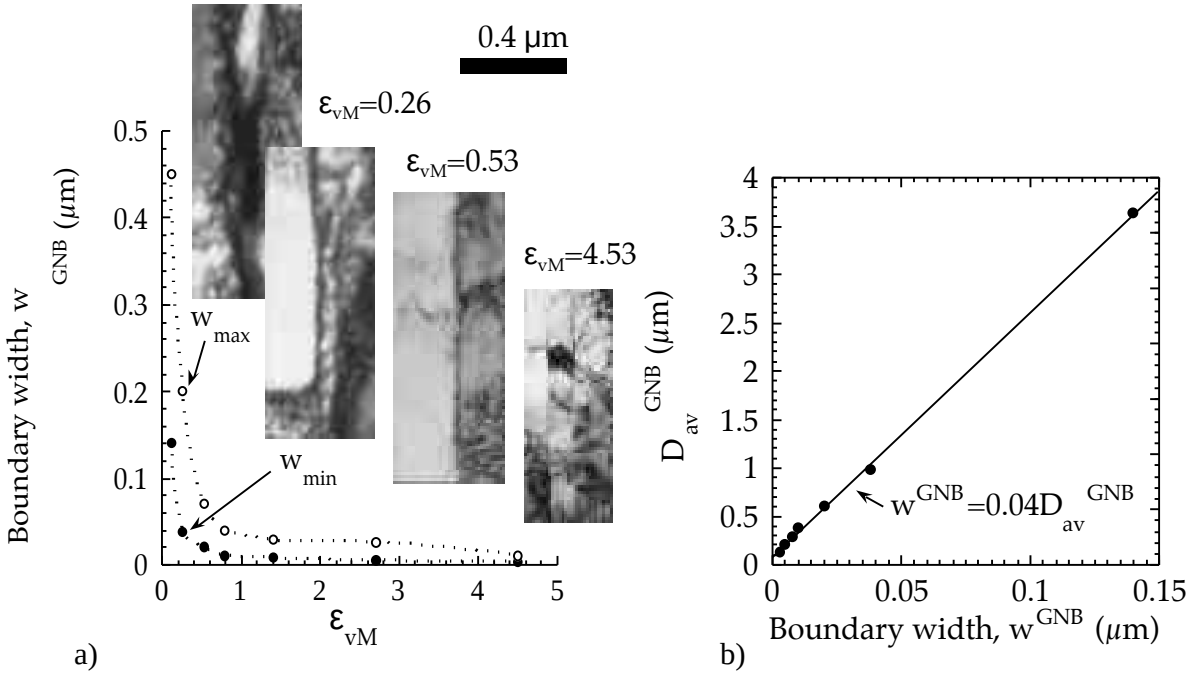

Fig. 11. The decrease in GNB width deccreases with increasing strain, mimicking the decrease in spacing. Note that a very narrow width of 3nm was observed at the largest strain.

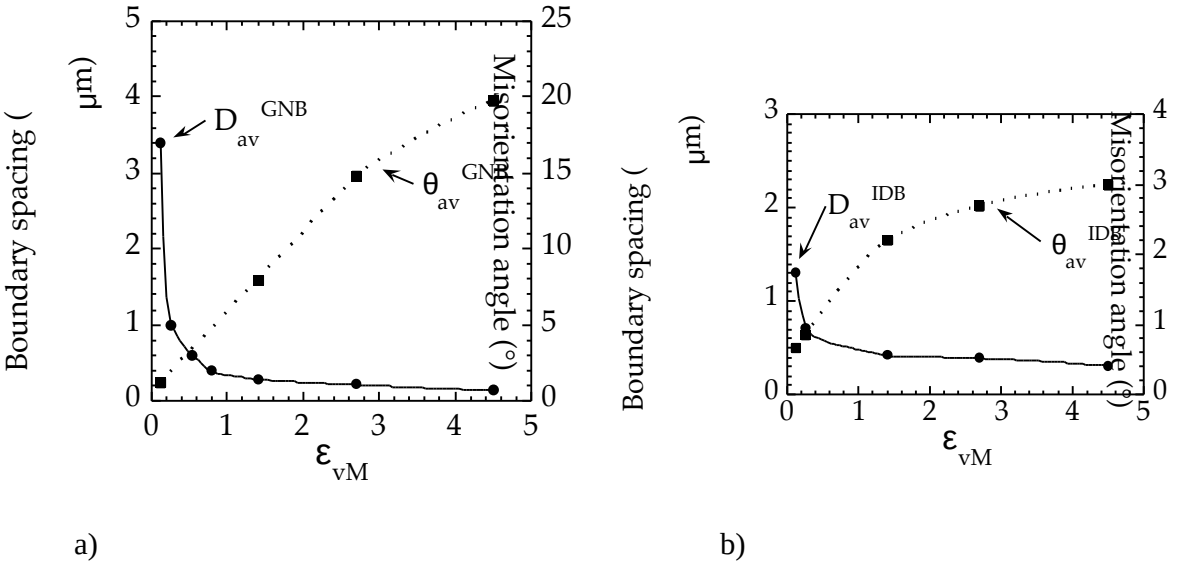

Fig. 12. Decrease in average boundary spacing and increase in misorientation angle across GNBs (a) and IDBs (b) as a function of strain. Note that neither the spacing nor the angle has saturated at large strain.

PREDICTING STRENGTH FROM MICROSTRUCTURE

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Motivation:
New tools for characterizing and analyzing microstructures have been developed in the last five years which create a firmer basis to explore the relationship between microstructure and flow stress. A central question regards the identification of the different microstructural elements, their relative strength contributions and persistence from small to large strain deformation.

Accomplishment:
A quantitative microstructural analysis was performed on pure polycrystalline nickel (99.99%) cold rolled to reductions including 10, 20, 70, 90 and 98% (ε_{vM} 0.1 to 4.5). Fig. 13 gives an example of the pattern of dislocation boundaries, IDBs and GNBs measured. Taking all the measurable structural parameters: spacing, misorientation angle, and dislocation density, and assuming additive strength contributions (eqn. 1), we derive from Hall-Petch and dislocation strengthening flow stress, σ, predictions (eqn. 2) which are in good agreement with the stress values and hardening rates observed experimentally (Figure 14).

$$\sigma = \sigma_0 + \sigma_{IDB} + \sigma_{GNB} \quad (1)$$

$$\sigma = \sigma_0 + M\alpha Gb\sqrt{(1.5S_v\theta/b)_{IDB}} + K_{HP}/\sqrt{D_{av}^{GNB(i)}} \quad (2)$$

In which σ_0 is the friction stress, M is the Taylor factor; α is a constant taken to be 0.24, b is the Burgers vector, G is the shear modulus, $(1.5S_v\theta/b)$ is a dislocation density based on the boundary area S_v and misorientation angle θ of the diffuse IDBs. In the second term the GNBs are treated like high angle boundaries K_{HP} is the Hall-Petch constant and $D_{av}^{GNB(i)}$ is a slip distance is related to the perpendicular spacing, D_{av}^{GNB}. No saturation of the calculated or experimental flow stress was observed. The changes in the hardening of Stages III and IV were captured quantitatively without invoking a change in mechanism.

Significance:
Scaling and similitude provide governing principles for structure formation. Based on this structural information and a detailed description of the morphology, structural parameters are identified, strength determining parameters chosen, and strength-structural relationships obtained. The suggestion is then made that two strengthening contributions should be considered: (i) dislocation strengthening due to the presence of low angle boundaries and (ii) grain boundary strengthening due to medium to high angle boundaries. The calculated individual strength contributions evolve differently with the strain and their addition leads to flow stress values and hardening rates in good agreement with those observed experimentally.

Publications:
D. A. Hughes and N. Hansen, "Microstructure and Strength of Nickel at Large Strains," *Acta Mater.*, 48 (2000) 2985-3004.

N. Hansen, X. Huang and D. A. Hughes, "Microstructual Evolution and Hardening Parameters," *Mater. Sci. and Eng.*, A317 (2001) 3-11.

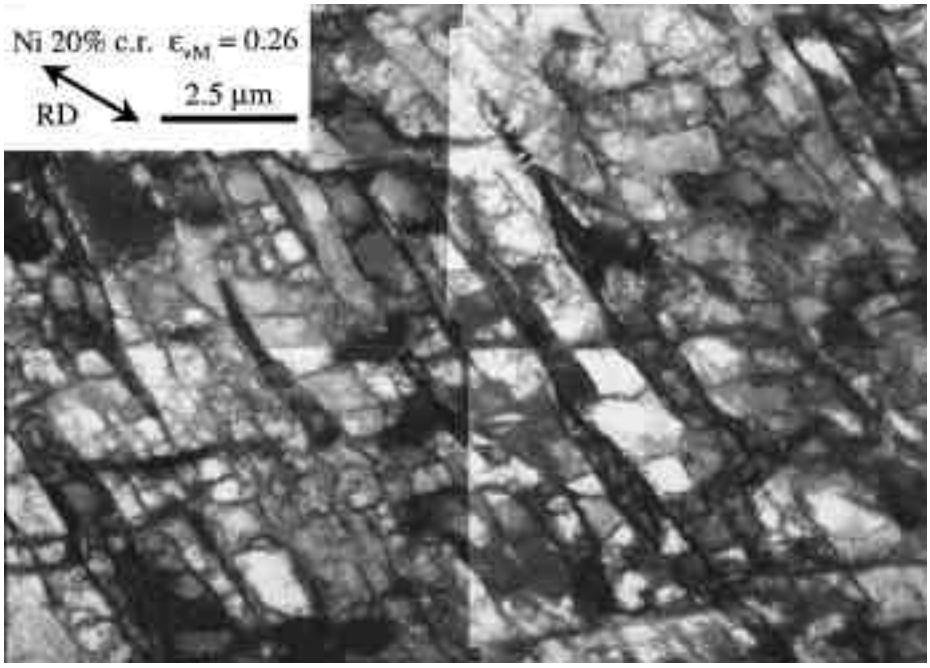

Fig. 13. TEM micrograph of cold rolled pure nickel illustrating nearly planar extended GNBs surrounding more equiaxed cells to form cell blocks.

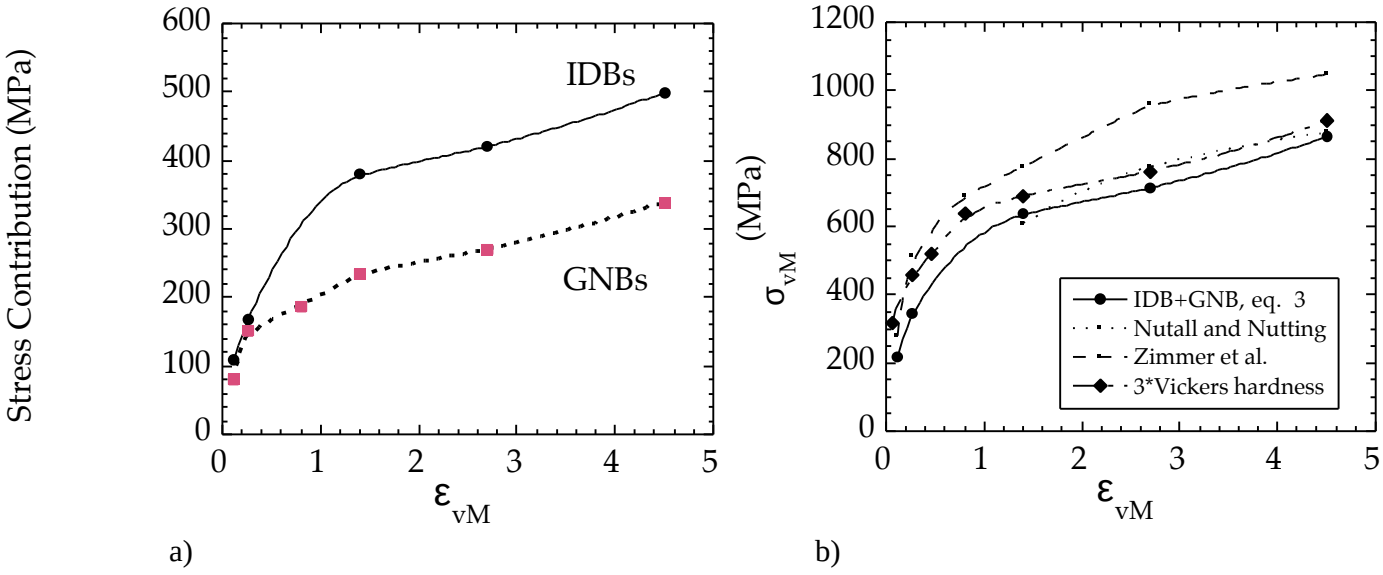

Fig. 14. a) The strength contribution of IDBs is based on dislocation strengthening, while that of the GNBs is based on Hall-Petch strengthening. Both types of boundaries contribute to linear hardening at large strains above 1.5. b) Stress predictions based on the combined microstructure match the level of hardness measurements on the same samples and stress strain data from the literature. Note in particular the agreement in the strain hardening that changes from parabolic at small strain to linear at large strain.

STACKING FAULT ENERGY AND MICROSTRUCTURE EFFECTS ON TORSION TEXTURE EVOLUTION

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[1]SNL/CA, [2]Univ. of Rosario, Argentina, [3]Univ. of California, Berkeley, [4]Brown Univ.

Motivation:
The formation of a preferred crystallographic texture during deformation of metals causes mechanical and magnetic property anisotropy. This formation is further modified by the concurrent microstructure evolution. Understanding the roles of dislocation slip versus deformation twinning in the formation of texture and microstructure are pertinent for the prediction and control of material anisotropy in metal industries from cars to magnets.

Accomplishments:
A series of experiments and simulations that vary the crystallographic texture and microstructure simultaneously were performed to establish the role of the microstructure in texture formation in fcc metals. The stacking fault energy of the metal (SFE) which is known to have a strong impact on texture and microstructure is the vital parameter used to make these variations. Torsion deformation was chosen to isolate the role of slip. It was determined that the wide variety of textures and microstructures observed as a function of SFE and temperature were developed by slip processes alone; twinning was not necessary, as previously thought (Figure 15). The different textures are caused by: i) variations in local slip patterns within a single grain as revealed by grain subdivision into differently deforming cell blocks and ii) more subtly by the cell block shape. The local selection of slip systems creating the lattice rotations within a cell block is altered by the planarity of slip. Slip planarity is controlled by the SFE and temperature. It is hypothesized that the newly observed texture components, that are distinct from the generally accepted ideal components, are created by the different slip processes occurring as a result of low SFE and low temperature. A more subtle effect of grain subdivision is related to the cell block shapes that develop as a function of SFE and temperature and correspond to the different textures observed. The shape of the cell block is related to the level of constraint required by the deformation. The slip pattern changes and cell block shapes correlate with the presence or absence of certain ideal texture components whose evolution is not simulated. Materials and conditions with similar deformation microstructures developed similar textures in the experiments.

Significance:
The different slip patterns formed within a single grain are at odds with the central Taylor assumption. How well the Taylor assumption is fulfilled on average depends on the SFE and deformation temperature as clearly shown by the development of new texture components. Future refinements of polycrystal plasticity simulations will need to include the characteristics of the microstructure. Clearly a grain cannot be considered as a homogeneous unit in realistic models of the deformation process.

Publication:
D. A. Hughes, R. Lebensohn, H. R. Wenk, and A. Kumar, "Stacking Fault Energy and Microstructure Effects on Torsion Texture Evolution," *Proc. Roy. Soc. London, A* **456** (2000) 921-953.

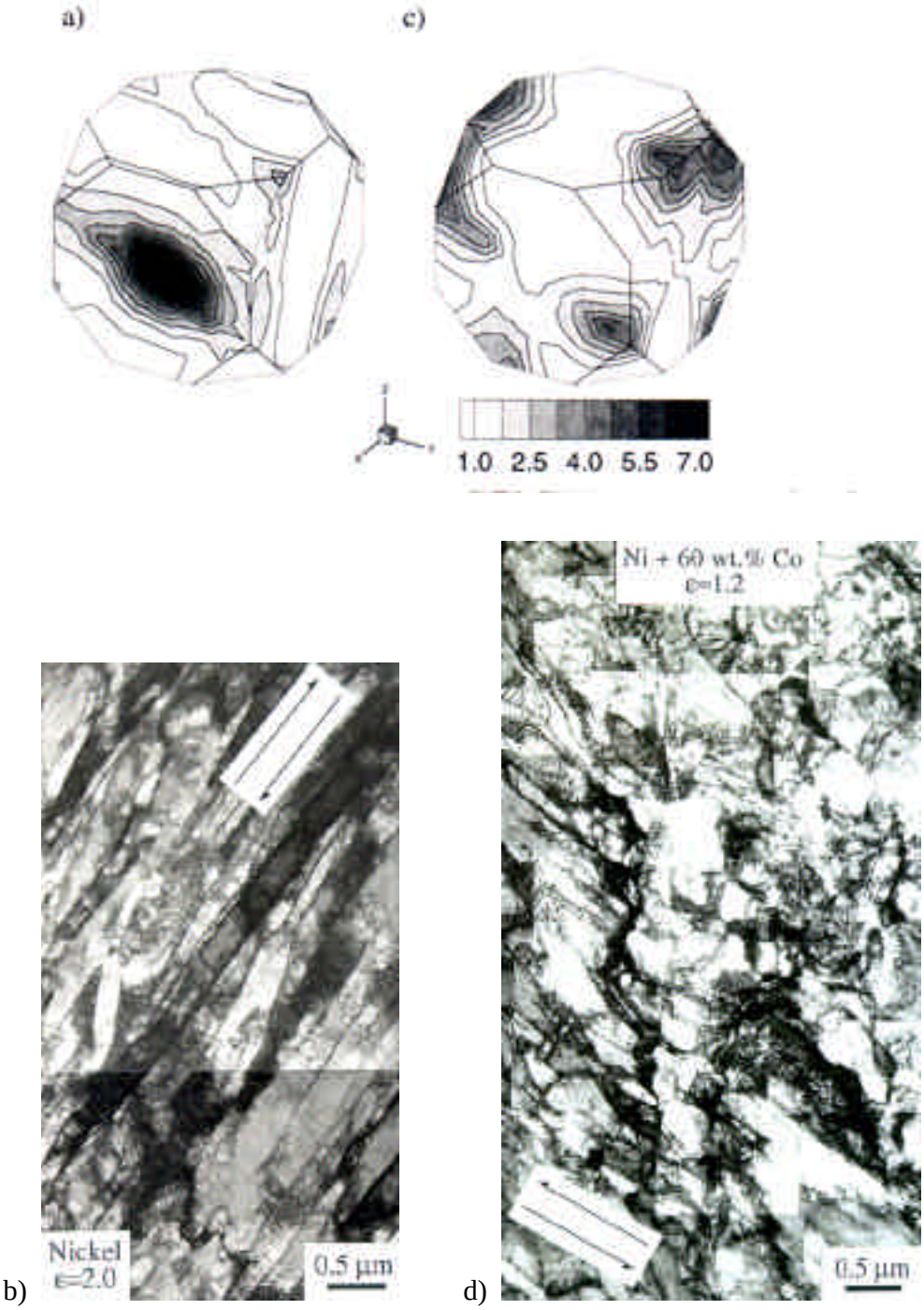

Fig. 15. a) Crystal orientation distribution function (CODF) plotted in the fundamental zone of Rodrigues space (Frank, 1988) showing a {100}<1$\bar{1}$0> preferred orientation (darkest shading) and b) a transmission electron micrograph of long cell blocks for nickel following torsion deformation to a von Mises effective strain ε=2. c) CODF showing a {$\bar{5}$42}<652> preferred orientation (darkest shading) and d) TEM micrograph of the more complex dislocation structure (few twins) for Ni+60 wt.% Co following torsion deformation to ε=1.2.

RECRYSTALLIZATION IN SUBDIVIDED DEFORMATION MICROSTRUCTURES

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[1]Sandia National Laboratories, CA [2]Risø National Laboratory, Denmark

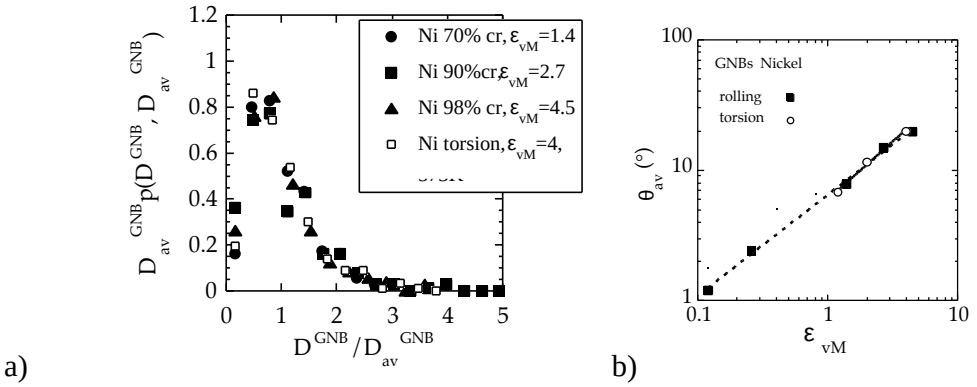
Motivation:
Grain subdivision by dislocation boundaries during the deformation of high purity fcc metals strongly influences the nucleation and growth of recrystallized grains. Direct connections are difficult to make for many reasons, including the need for fine scale data over very large areas or volumes. However advanced experimental techniques combined with structural analyses are providing new insights.

Accomplishment:
We consider dynamic recrystallization at low homologous temperatures in a deformed structure which is well characterized. Detailed TEM studies are facilitated by the wide spectrum of sizes and high density of nuclei and/or potential nucleation sites formed at the beginning of dynamic recrystallization. Our previous structural analyses, on typical distributions of boundary misorientation angles and spacings that develop during deformation, provide probes to identify the local heterogeneities associated with nucleation. Nucleation in these samples ocurred in clusters of multiple orientations at grain boundaries and intragranularly in the deformed structure, as well as in regions with localized shear occurred. The neighborhood around 23 new nuclei was investigated using local orientation measurements in the TEM. These measurements showed that 80% of the nuclei had at least one high angle boundary segment. Some also had a low angle boundary that connected them to the deformed matrix. Nuclei formed in regions with a significantly higher number of high angle boudnaries than the average structure.

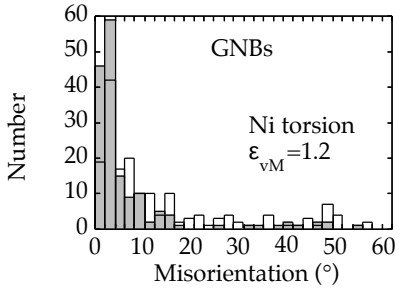
Significance:
Deformation produces a structure with an intermixture of high and low to medium angle boundaries. During recrystallization these high angle boundaries produced by the deformation can become nucleation sites. Low temperature dynamic recrystallization, in which little recovery occurs prior to recrystallization, provides a means to directly and quantitatively explore the relationship between the deformed structure, nucleation and subsequent growth. These relationships are necessary as input into recrystallization models.

Publications:
D. A. Hughes, and N. Hansen,"Recrystallization in Subdivided Deformation Microstructures," Proceedings of the First Joint International Conference on Recrystal-lization and Grain Growth, ed. G. Gottstein and D. A. Molodov (Aachen: Springer Verlag, 2001) 659-668.

D. A. Hughes, "Proceedings of the 21st Risø International Symposium: Recrystallization - Fundamental Aspects and Relations to Deformation Microstructures, ed. N. Hansen, et al. (Roskilde, Denmark: Risø National Laboratories, 2000), 49-70. *Journal of Surface and Interface Analysis*, 31, (2001) 560-570.



a) b)



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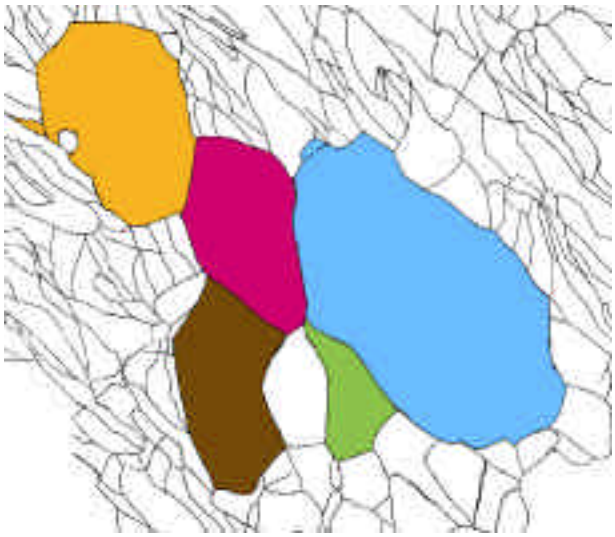

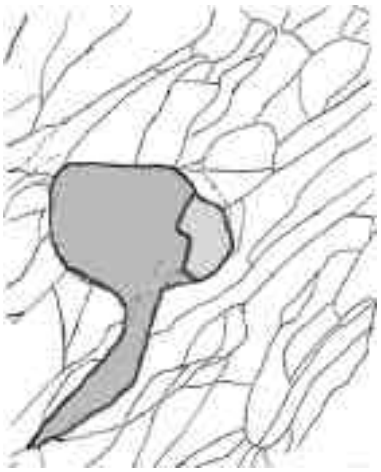

d) e)

Fig. 16. a) Scaled spacing distributions for deformed strucutres in rolling and torsion. b) Power law relationship between angle and strain. c) GNB misorientation angle distribution for the average deformed structure, stippled, compared to areas near nuclei, clear, which have more high angle GNBs d) Tracing of cluster of intragranular nuclei with different orientations from TEM image 8μm hi gh. e)Tracing of TEM image of potential nuclei arising from a high angle lamellar band (shaded). Annealing twin is lighter shading. Image is 5.5 μm high.

BIMETALLIC LAYERS IN CU-AG EUTECTICS

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Motivation:
Technologically, the development of a fine layered Cu-Ag eutectic is desirable for high strength, high conductivity applications. Current methods for constructing this layered structure, such as, vapor deposition and Taylor wire, can produce only small specimens and are expensive to develop and maintain. The ability to produce a very small-scale layered structure by mechanical deformation would enable much larger products to be more efficiently produced. This process also enables us to simulaneously examine changes in the deformation mechanism due to the presence of two phaeses. Most of the previous research on these Cu-based eutectics has focused on quantifying the strength increases due to various production techniques and deformation conditions. Other than brief qualitative assessment of micrographs, little work has been done to quantitatively characterize the structures and textures that develop during these deformation processes and directly contribute to the increase in strength.

Accomplishments:
The microstructural evolution of the Cu-Ag eutectic during rolling deformation has been characterized(Fig. 17a and b). Cold-rolling of the Cu-Ag eutectic to 97% reduction produced a layered structure with ribbons of copper in a silver matrix and an average layer spacing of approximately 20 nm (Fig. 19). At intermediate strains, many finely spaced boundaries form in the silver phase while little internal boundary development is observed in the copper phase. At 97% reduction, the microstructure has been reduced to the aforementioned nanoscale layered structure with relatively little internal dislocation or boundary content. The partitioning of the deformation between the copper and silver phases has been interpreted with respect to the spacing and misorientation of boundaries formed within the individual phases and the increasing misorientation at the copper/silver interfaces. Local orientation measurements indicate that the alternating Cu-Ag layers cooperatively deform.

Significance:
There is little available literature on the behavior of eutectic systems during mechanical deformation. This research provides some of the first real insight into the evolution that occurs during deformation of eutectic systems. While the microstructural evolution of the Cu-Ag eutectic and single phase alloys (Cu,Al,Ni) during deformation have some parallels, there are notable differences. specifically the differences in boundary spacing (Fig. 19) and misorientation evolution of the distribution of Cu-Ag boundaries vs. internal Cu-Cu or Ag-Ag boundaries indicates that deformation of the eutectic can not be treated as simply the deformation of two independent phases.

Publications:
M.T. Lyttle and D.A. Hughes, “Nano-lamellar Structures in a Rolled Cu-Ag Alloy”, Material Instabilities and Patterning in Metals, ed. H. M. Zbib, et al. (Warrendale, PA: MRS, 2001) MRS Proceedings Volume 683E, BB.1.4.1-6.
M.T. Lyttle and D.A. Hughes, “Layered Nanoscale Structures in a Cold Rolled Cu-Ag Eutectic” in preparation.

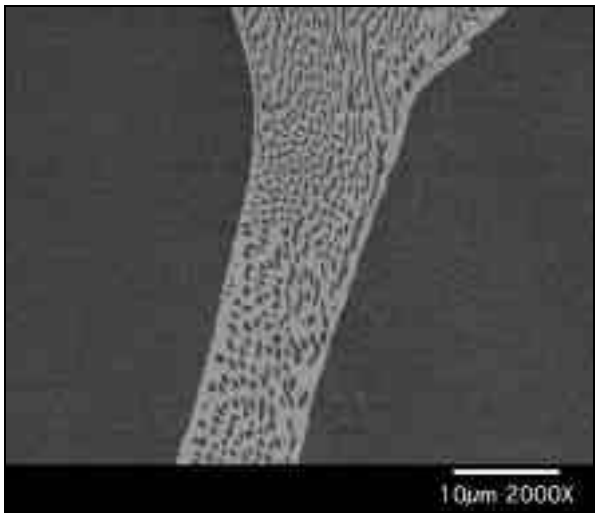
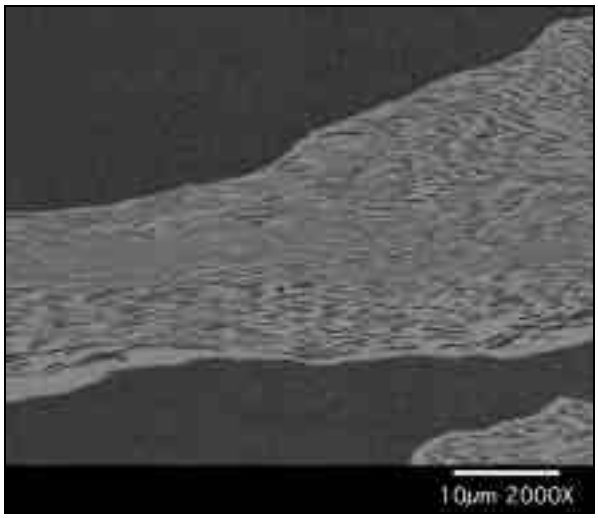


Fig. 17a. SEM micrograph of as-cast Cu-Ag eutectic structure; dark areas are copper, light area is silver.



b. SEM micrograph of Cu-Ag eutectic after 70% reduction; dark areas are copper, light area is silver.

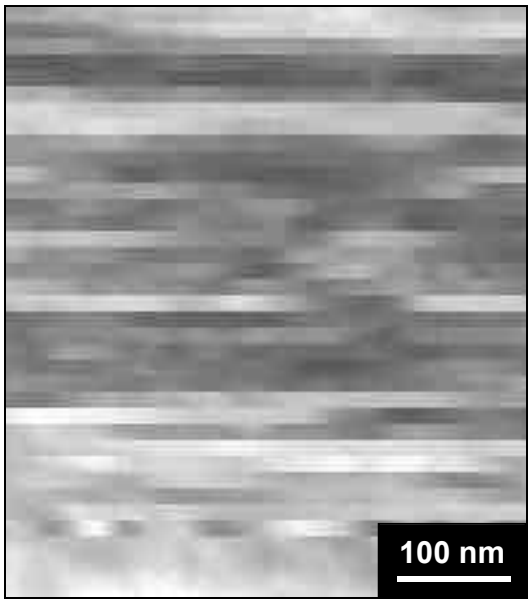


Fig. 18. TEM micrograph of Cu-Ag eutectic rolled to 97% reduction. Structure is primarily alternating layers of copper and silver.

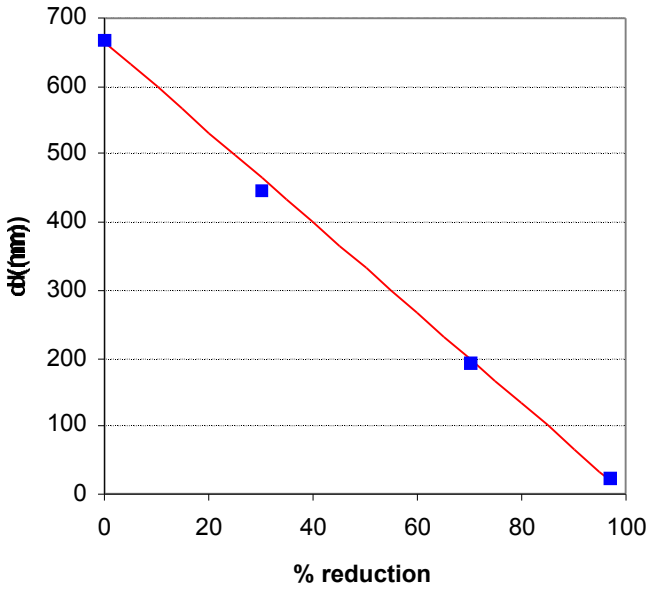


Fig. 19. Decrease of boundary spacing with amount of rolling reduction. Fitted line represents the decrease produced by geometric thinning of an initial boundary spacing of 665 nm.

SPATIAL ORDER AND THE EVOLUTION OF DEFORMATION-INDUCED STRUCTURES

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Motivation:
Deformation of metals and alloys typically leads to a complex system of thin, dislocation-rich boundaries separating nearly dislocation-free regions (Fig. 20). This boundary arrangement determines material properties, with mechanical strength closely relating to the characteristic spacing between boundaries. A model for the evolution of the boundary spacing distribution with increasing strain would therefore provide a formalism to predict properties based on this evolution.

Accomplishments:
The arrangement of experimental boundaries reveals short-range order. Additionally, experimental observations show that boundary spacing distributions $P(D,\varepsilon)$ at different strain levels collapse to a universal distribution when each distribution is scaled by its average spacing, D_{av} (Fig. 21a). We exploit these features and the strain invariance to develop a model of the evolution of the boundary spacing distribution. The goal is to predict locations and rates at which new boundaries are formed and old boundaries are removed. Consider that $P(D,\varepsilon)$ evolves according to:

$$dP(D,\varepsilon)/d\varepsilon \approx + \int_D^{\infty} dy\, P(y,\varepsilon)\, r_{CR}(y-D,D,\varepsilon) - P(D,\varepsilon) \int_0^{D} dy\, r_{CR}(D-y,y,\varepsilon), \quad (1)$$

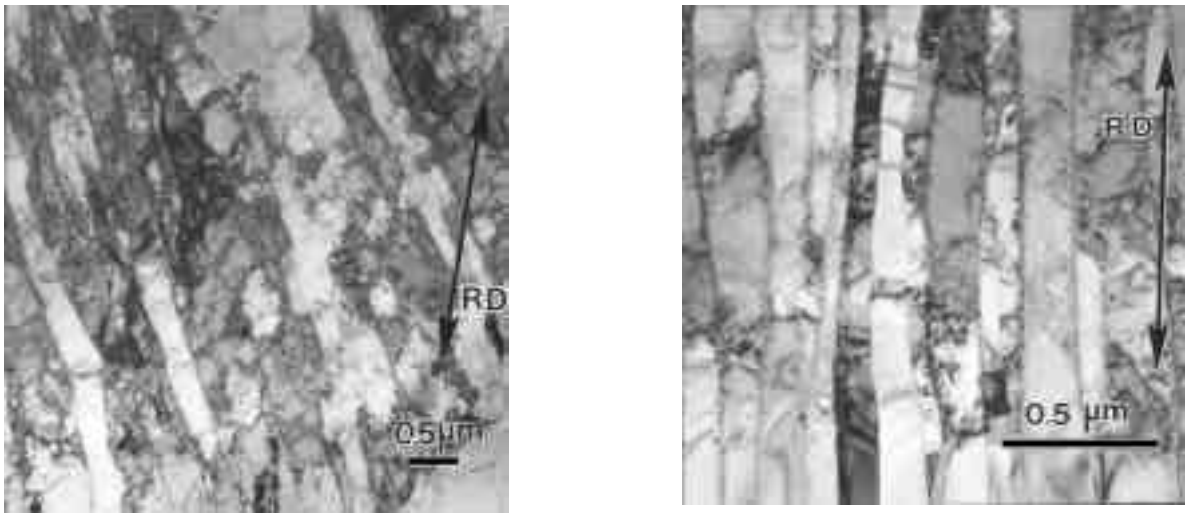
with $P(D,0)=0$, where $r_{CR}(y_1,y_2,\varepsilon)$ describes the odds of creating a boundary which splits the spacing y_1+y_2, between two adjacent boundaries, into two new spacings, y_1 and y_2. If one chooses the simplest function $r_{CR}(y_1,y_2,\varepsilon)=(y_1+y_2)^{\alpha}$, that is not dependent on strain, then integration of equation 1 yields the approximate analytical expression:

$$P(\zeta,\omega) \approx c\omega\zeta^{\alpha+1}\exp(-\omega\zeta^{\alpha+1}), \quad (2)$$

with $c=(1/D_{av})(1+\alpha)^2\omega^{1/(\alpha+1)}/\Gamma[1/(\alpha+1)]$, $\zeta=D/D_{av}$, and $\omega=\varepsilon D_{av}^{\alpha+1}$. Equation 2 is only independent of the strain if $\omega=\varepsilon D_{av}^{\alpha+1}$ is a constant. This constraint is checked in Fig. 21b showing that this strain dependence of D_{av} is observed. The strain dependence also suggests a value of α which is 1/2. This choice of α weights the creation of a new boundary between adjacent boundaries by the square root of the spacing thereby providing an excellent fit of the scaling function to the experimental data, Fig. 21a.

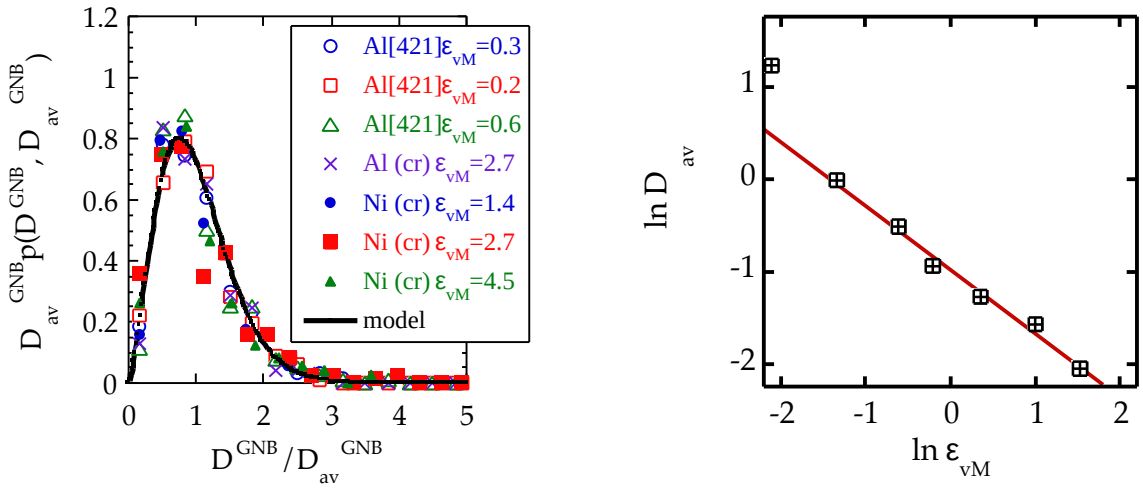
Significance:
The success of this statistical model in describing the results, despite the complexity of boundary formation, provides us with a new and rational means to construct models of microstructure. Such models provide a basis for discussion of how mechanical properties can be engineered efficiently within the many high volume industries associated with the deformation processing of metals and alloys.

Publication:
M. C. Bartelt and D. A. Hughes, "Spatial Order and the Evolution of Deformation-Induced Structures," submitted for publication, 2001.



a) b)

Fig. 20. Extended dislocation boundaries following different amounts of cold rolling nickel look similar when the magnification is scaled by the average spacing, D_{av}. a) following 37%cold reduction, b) following 95% cold reduction



a) b)

Fig. 21. a) Universal distributions for cold rolled nickel and compressed aluminum single crystals showing the model fit, eqn.2, based on α=1/2 derived from the experimental data in b) for nickel, b) Strain dependence of D_{av} for cold rolled nickel showing a power law relation.

FACTORIZATION OF CORRELATIONS IN DISLOCATION STRUCTURES

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Motivation:
At sufficiently high strains, extended dislocation boundaries formed during deformation organize into fine lamellar-like structures; see Figs.22(a)-(b). The minimum set of dynamical rules that support such order has not been identified, nor have conventional models of (many-) dislocation interactions been able to reproduce it. A different way to approach these issues is to develop master equations for the evolution of the distribution of linear spacings (or "gaps") between the extended dislocation boundaries, accounting for such processes as nucleation of new boundaries, coalescence of nearby pairs of adjacent boundaries, or removal of boundaries due to crystal rotations into coincidence, as suggested by experiment. Difficulties arise because these equations couple to a full hierarchy of spatial correlations. For instance, the rate of coalescence of pairs of adjacent boundaries, which affects the probability of finding a pair of boundaries at a certain distance (or a gap of a certain size), depends on the probability of finding triplets of adjacent boundaries (or pairs of gaps). Consequently, the ease with which these evolution equations can be solved relies on some knowledge of the form of these higher-order correlations, and then on the ability to truncate that hierarchy at some level, using factorization of the joint probabilities. It is thus important and useful to verify to what extent such property holds for experiment.

Accomplishments:
Fig.22(c), shows data for the joint probability (averaged over many adjacent pairs of boundaries), $P(d_1,d_2)=P(d_2,d_1)$, of finding two adjacent gaps (or spacings) of linear size d_1 and d_2, measured perpendicular to the extended boundaries in Fig.22(b). Clearly, one is unlikely to find gaps between boundaries much larger than the average spacing, D, con-sistent with the expectation that new boundaries are likely to form in those (plastically soft) regions during deformation. Consequently, there are also very few pairs of large adjacent gaps between boundaries. Detailed inspection of cross-sections of the data for the joint probability $P(d,d_2)$ versus d_2, for selected d_1 values, reveal sub-distributions that look very much like the single-gap function $P(d_2)$. This result suggests that, to good approximation, the relation $P(d_1,d_2)\approx P(d_1)P(d_2)$ is satisfied in experiment, i.e., on average the sizes of two adjacent gaps between boundaries do not depend strongly on each other; see also Ref.1 for misorientation-spacing correlations. That result holds in particular when one of the gaps is small (compared to D), as is relevant for boundary coalescence; Fig.22(d).

Significance:
This is a powerful result: the higher-order spatial correlations are approximately determined given the form of the single-gap function, P(d). We are currently further testing this feature in simulations of the dislocation boundary formation process. Here we can directly compare full separation distributions of dislocation boundaries generated using the joint probabilities, $P(d_1,d_2)$, with those generated using the factorized probabilities, $P(d_1)P(d_2)$.

Publication:
D.A. Hughes, Distributions of Low and High Angle Boundaries in Deformed Metals, in Recrystallization '99, Proceedings of The Fourth International Conference on Recrystallization and Related Phenomena, edited by T. Sakai and H.G. Suzuki (Sendai, Japan: JIMIS, 1999) p.111-118.

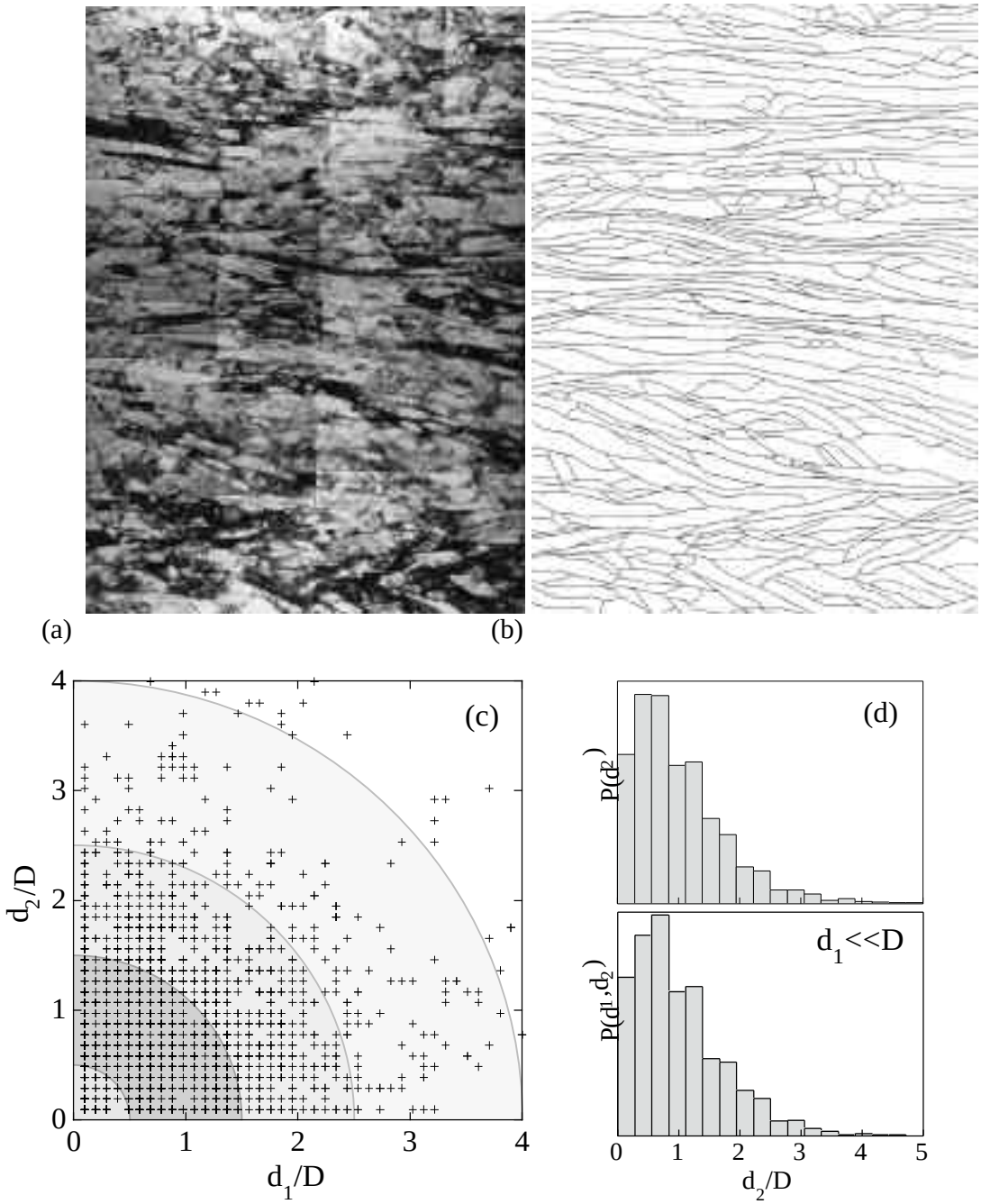

Fig.22. TEM image ($6.5\times9\mu m^2$) of a high-purity (99.99%) Ni polycrystal (initial grain size ~100μm) deformed by cold-rolling to 90% reduction ($\varepsilon_{vM}\approx2.7$). The image shows contrast from a configuration of extended boundaries nearly parallel to the rolling direction (11° off the horizontal), as is typical at this strain. They are on average $D\approx0.2\mu m$ apart, and each accommodates an average crystal misorientation of 15°. They enclose blocks of cells of lower dislocation density and different characteristic scale. In (b), we traced only the extended boundaries. (c) The symbols are observed pairs of adjacent gaps between boundaries, and the shaded regions give a coarse-grained projected view of the value of the average joint probability, $P(d_1/D,d_2/D)$, for adjacent gaps (darker shade has higher value). (d) $P(d_1<<D,d_2)\approx P(d_2)$, which is relevant to describe coalescence of nearby boundaries.

STRESS PATTERNS OF DEFORMATION INDUCED PLANAR DISLOCATION BOUNDARIES

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Motivation:

During deformation dislocation boundaries are created whose spacing is inversely proportional to the stress. To model the structural evolution and thus stress evolution, the question arises as to where in the existing structure a new boundary will arise. One idea is that the self stress fields of existing boundaries govern this process. Computing these stress fields is difficult however, because, one needs to consider finite boundaries composed of 3 independent Burgers vectors in a representative volume. Analytical solutions do not exist and periodic boundary conditions cannot be applied for general and realistic cases.

Accomplishments:

A multi-scale model coupling discrete dislocation dynamics (DD) with continuum plasticity and finite element analysis (FEA) is used to study the self-stress field of geometrically necessary (dislocation) boundaries (GNBs) Figs. 23 and 24. The results for a single GNB are presented here. The internal structure of the GNB is obtained from Frank's formula using the experimentally measured misorientation angle/axis pair as the input. Several different types of model boundary conditions (using FEA) are analyzed together with the effect of different parameters like the domain length and mesh sensitivity. It is shown that the boundary conditions for the FEA strongly affect the predicted internal stress fields of these dislocation boundaries, particularly the long-range effect, Fig. 25. The best boundary condition was selected by first simulating cases for which comparisons could be made with an analytical model.

Significance:

A technique has been developed in which we can study the internal stress fields and interactions of dislocation boundaries with each other, and applied stress and with gliding dislocations. This step is important in obtaining governing mechanisms for dislocation boundary creation and spacing evolution.

Publications:

S. M. A. Khan, H. M. Zbib, and D. A. Hughes,"Stress Patterns of Deformation-Induced Planar Dislocation Boundaries," Material Instabilities and Patterning in Metals, ed. H. M. Zbib, et al. (Warrendale, PA: MRS, 2001) Volume 683E, BB.3.8.

D. A. Hughes, S. M. A. Khan, A. Godfrey, and H. Zbib, Internal Structure of Deformation Induced Planar Dislocation Boundaries," *Mater. Sci. Eng. A.* 309-310 (2001) 220-226.

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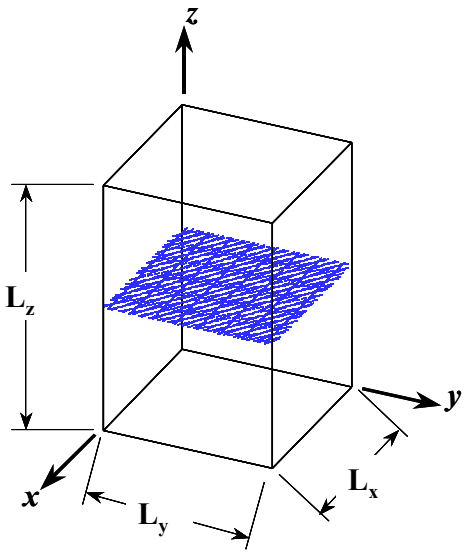

Fig. 23. Problem configuration showing the computational cell and the dislocation boundary.

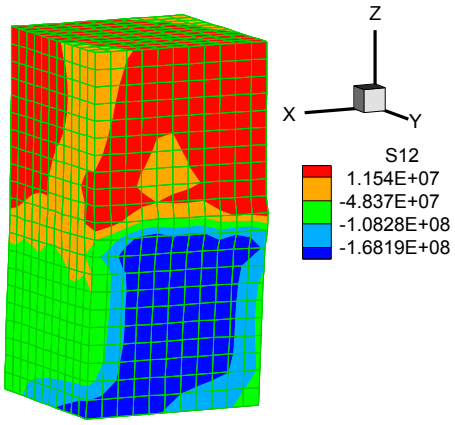

Fig. 24. Stress contours: deformed crystal only one base fixed with σ_{12} contour shown (displacements are magnified10X)

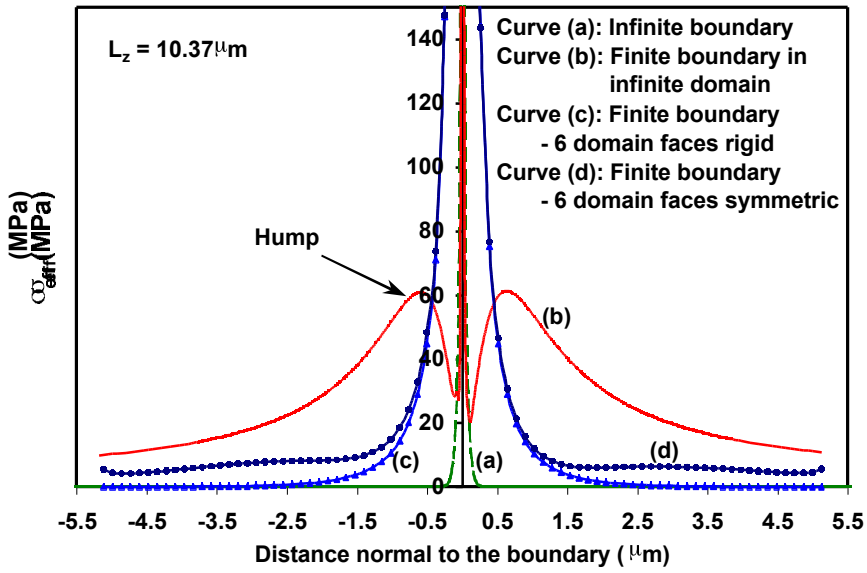

Fig. 25. Distribution of effective stress, multiscale modeling vs. DD .

Proposed Work

Experimental

The graded nanostructures we have produced by sliding deformation enable us to explore the plastic behavior of single phase metals at a fine scale not possible before. An order of magnitude decrease in size is realized by adapting this friction process compared to other bulk deformation processes (e.g. rolling, equal channel angular pressing, drawing, and accumulative roll bonding (Mishra *et al.,* 2000). This experiment also provides the unique opportunity to examine an enormous range of size scales and strain levels at once in a single sample.

We have demonstrated the remarkable level of scaling from 10,000 to 10nm, of the GNBs. This advance brings us into a scale accessible by atomistic calculations (Schiotz *et al..*, 1998; Rigney and Hammerberg, 1998; Van Swygenhoven and Caro, 1997 and1998; Kebliski *et al.*, 1999; Yamakov *et al.*, 2001). These findings, which show that the deformation mechanisms are continuous over this range of scale are pertinent to the ongoing discussion of transitions in yield and dislocation behavior at small scales, (*e.g.*, Scattergood and Koch, 1992; Embury and Hirth, 1994; Chu and Barnett, 1995; Sanders *et al.*, 1997; Anderson *et al.*, 1999; Misra *et al.*, 1999).

To determine the lower limits of this scaling and a transition in behavior, we need to create GNB/layer spacings less than 10nm. To accomplish this goal, a new piece of equipment has been designed and built which enables us to perform our sliding experiments at different temperatures, with different normal loads, sliding rates and counterface materials. Large normal pressures and short sliding distance distinguish this process from typical friction and wear tests in which plasticity has been examined as a wear and friction mechanism (see especially the wear and microstructure work of Rigney and Hirth, 1979; Heilmann *et al.*, 1983; Rainforth *et al.*, 1992)

In addition to the those test parameters, solid solution alloying additions to pure fcc metals, e.g. Fe or Ag added to Cu can be used to further reduce the size scales and to inhibit the recovery and or recrystallization of the structure. We hope to achieve size scales less than 5nm with the possibility that amorphous layers may also form (Bednar and Kuhlmann-Wilsdorf, 1995). These studies are complementary to other groups that are looking at bimetallic laminates formed by a Taylor wire method, or vapor deposition, see for example (Han *et al.*., 1998; Misra *et al.*, 1999).

Many details regarding these structures are unknown and need to be determined. The average spacing and spacing distributions have been measured for GNBs. These boundaries separate crystal volumes that are rotated with respect to one another. We know that from diffraction spot patterns that the extended boundaries provide a spatially intermixed distribution of low 4° to high >15° angle boundaries, but the local orientations between them, and the exact boundary misorientation angles and their distributions have not been determined using Kikuchi pattern analysis for fine average structures <100nm. We want to know if the trends in angle distribution and the development of preferred textures observed at spacings >100nm for monotonic deformation will continue as smaller sizes are obtained. This is an issue which has been taken up at larger size scales for non-monotonic types of large strain deformation such as equal channel angular extrusion, accumulative roll bonding, and cyclic extrusion compression (Mishra *et al.*, 2000).

The defect structure between the boundaries is clearly resolved for spacings above 30 nm. While much of this volume is nearly dislocation free, short dislocation boundaries and a few isolated dislocations are observed bridging the flat extended boundaries (Fig. 8). The short bridging dislocation boundaries are low angle boundaries. Additionally a few deformation-

induced twins are observed (Fig. 8) as well as unresolved features bridging the narrowest boundary spacings. These latter features may be stacking faults. These structures need to be quantified with different TEM imaging techniques to probe the origin of the diffraction contrast when spacings are less than 30nm, see for examples the fringe contrast in Fig. 8. Tilting experiments and weak beam dark field techniques are necessary although the fineness and apparent "complexity" of the overall structure make this a challenge (e.g, Forwood and Clarebrough 1991).

The detailed measurements, from the crystallographic texture that is formed to the configuration of dislocations (or their lack) yield important information on the dislocation slip processes possible at these fine scales. Additionally, the limited ductility and early fracture frequently observed in nanostructured metals and alloys is a persistent problem (*e.g.*, Sanders *et al.*, 1997). Limited ductility is related to not only internal flaws such as porosity, but also the internal structure within and between the boundaries. What defects or structures contribute to the limited ductility can be explored by recovery experiments at higher temperatures. Recovery experiments will be done in-situ in our TEM heating stage or ex-situ in bulk samples.

Optimization of strength and ductility is often achieved by warm deformation in which recovery occurs simultaneously with the deformation (*e.g.* McQueen 1977). Warm deformation is defined as that deformation occuring roughly from 0.3 to 0.5 of the melting temperature. Very little detailed work has been done on the microstrucutral mechanisms of recovery (e.g. Longo and Reed-Hill 1974; McQueen 1977; Prinz *et al.*, 1981). Interest however, has been rekindled in light of the modern microscopy techniques that are available, e.g.the detailed work on symmetric single crystals of aluminum (Huang and Humphreys, 2000a 2000b). For our program both friction and torsion experiments will be performed in this warm deformation range. The latter is a continuation of the dynamic recrystallization work that has just begun to investigate the early stages of nucleation and the conditions that enable the recovery of dislocation and high angle boundaries (see also Jones *et al.*,). with respect to the former, nanocrystalline materials can undergo primary and secondary recrystallization at room temperature, often producing a structure of micron-sized grains in a finer-grained matrix. This coarsening process influences subsequent deformation behavior. Again our friction experiments provide us with structures in which to analyse this phenomena and to compare with the processes occuring at larger scales in torsion.

To study transitions in the misorientation angle distribution, mechanical experiments will be performed that attempt to modify this transition in pure nickel. Nickel is chosen as a model material because a high density of GNBs can be obtained and the dislocation structure is stable and of a size scale optimal for TEM observation. For cold rolling of medium to high stacking fault energy metals like nickel, changes in the scaled misorientation angle distribution occur at a deformation level from 60 to 70% cr (1 to 1.4 ε_{vM}). This result is observed for samples without a strong initial crystallographic texture.

It is believed that this transition is related to the accumulation of lattice rotations as preferred textures are created. Three possibilities will be explored that make use of this hypothesis. First, significantly different starting textures will be used for deformation by monotonic rolling. It is known from single crystal and polycrystal studies at lower strains, that the average GNB angle and spacing depend on the initial crystal orientation at small strains (e.g. Liu and Hansen, 1995; Driver *et al.*, 1995; Huang and Hansen, 1997). Second, monotonic torsion will be used to produce different preferred textures. Preliminary evidence indicates that high angle boundaries may form with a different distribution shape than the bimodal shape observed in rolling (Hughes and Hansen, 1997). Third, cyclic torsion will be employed. Large strain amplitudes of 0.2 to 0.4 will be used, so that a well formed dislocation structure is produced in

the first cycle. Note that this moderately high strain amplitude spans the gap between strain amplitudes used in fatigue testing and those generally employed by equal angular channel die extrusion (Segal, 1995; Agnew *et al.*, 1998). Short thin-walled tube torsion samples and rolling followed by tension will be utilized as the best means to determine the flow stress and strain hardening rate for each of these cases. While temperature and strain rate are traditional variables that could be explored to induce different behavior, we will focus on ambient temperature and moderate strain rate tests. Rolling strain rates are about $3s^{-1}$, whereas torsion will be performed at somewhat lower rates of $0.01s^{-1}$. The strain rate sensitivity of nickel under these conditions is extremely low.

For characterization of the structural evolution, observations of the structure will be made following total strain levels close to the known transition at 1 to 1.4 ε_{vM}. Somewhat lower and higher strains will also be probed so that trends in the evolution of the misorientation angle distribution shape can be assessed. The microstructures that develop will be quantitatively characterized in a field emission TEM using a fast semi automatic Kikuchi pattern analysis for orientation measurements. Both high resolution and large area, i.e., multi-grain regions, may be measured with this technique in a single sample. Supplemental characterization at much larger scales will be made with back scattered scanning electron microscopy and EBSP techniques for orientation measurements. Data gathered from these measurements will include dislocation boundary spacings, boundary misorientation angles, and boundary plane and morphology for both IDBs and GNBs, as a function of the applied deformation, material, and texture. Crystallographic texture will be determined from the EBSP measurements and neutron diffraction. Information on boundary spacings, neighbor correlations, and angle correlations will be obtained from these tests to aid the theoretical studies.

Comparisons are underway between these “monomaterial” results from rolled fcc nickel and friction deformed copper, with those in an fcc bimetal. A copper-silver directionally solidified eutectic (c.f. Figs. 17 and 18) was rolled to create regions with Cu-Ag layers and Cu-Cu GNB layers. Laminate structures developing within the eutectic regions are observed to evolve with a different pattern of boundary spacing and misorientation angle distributions than in a single phase sample. The continued development of these structures and parameters at larger strains will be examined by rolling to 99% reduction (ε_{vM}=6) and by friction deformation. Local orientation measurements indicate that the deformation ocurrs cooperatively between neighboring Cu and Ag layers. The nature of this cooperative deformation needs to be explored through polycrystal plasticity and slip pattern modeling (e.g. Winther *et al.*, 2000).

These phase boundaries appear to be very good sinks for the dislocations associated with the deformation, as few dislocations and boundaries are observed within the laminates. In depth TEM analysis of these highly deformed laminate boundaries, and any dislocations associated with them, will provide a new extension of the research performed with respect to the dislocation interactions of laminates formed by vapor deposition (*e.g.*, Han *et al.*, 1998) or in strained epitaxial layers (Stach *et al.*, and Hull *et al.*, 1999). As in the single phase case, the contrast structure inside the the bimetallic nano-laminates as well as the nature of the Cu-Ag boundaries need to be identified through TEM experiments. The dislocation structure and the associated scaling of parameters in the copper rich regions have yet to be fully measured. They may or may not resemble those observed for rolling of pure fcc metals. In either case, we will obtain further insight into the formation of layered structures during deformation.

The structural characterization is used to model the deformation process and mechanical properties. Thus, tensile strength measurements will be performed on the exact eutectic compostion deformed to several rolling reductions. This data combined with the microstructural

characterization should allow a finer consideration of the role in strengthening due to the interaction of the copper and silver phases in the eutectic.

Theoretical

Our theoretical effort will focus on one key aspect: understanding the selection, at low strain, and the evolution, at higher strain, of the GNB and IDB spacings. This study should then yield specific predictions regarding the possibility of transitions in the shape of the scaling function for the spacing distributions at higher strains, for different experimental structures and a range of laminate scales.

The formation of GNB and IDB structures during plastic deformation combines three basic processes: the *nucleation* of new boundaries due to pinning of dislocations, at defects or following dislocation-dislocation reactions; the *growth* and hardening of formed boundaries due to continued trapping of gliding dislocations; and the *coalescence* of contacting boundary segments at higher strains. It is interesting to note that the characteristic length scale for trapping and multiplication of dislocations is of the order of 2.5nm (using a shear modulus of 10^5MPa, an effective flow stress of 500MPa, and Burgers vectors on the order of a lattice constant). This value is comparable to the average spacing between dislocations forming an IDB of 3°average misorientation angle measured for the above conditions. However, this value is much smaller than the typical spacings between all types of dislocation boundaries observed at this condition (cf. Fig. 2d). The selection of the GNB spacings is clearly a more complex function of the different parameters, yet a certain progression of average spacings is consistently observed. To aid in this theoretical work, we plan experiments that will examine spatial and temporal correlations between dislocation content, slip system activity, boundary width, misorientation angle and spacing in order to derive underlying mechanisms for the observed scaling behavior.

As noted above, competition between GNB formation and coalescence must be taken into account at higher strains. Clearly, coalescence of GNBs is not guaranteed to produce noticeable differences in the shape of the scaling function for GNB spacings. The outcome appears to depend strongly on details of the coalescence process, again based on results for simple one-dimensional models (Godfrey and Hughes, 2000). It should also reflect correlations in the spacing of neighboring GNB pairs, which develop during deformation.

In addition we will model the dislocation content of GNBs and IDBs. The purpose of this modeling effort is to examine the self stress fields, stability and interactions between multi-Burgers-vector dislocation boundaries to obtain information on boundary creation. We will use experimental data as we generate them, but we will also build boundaries out of simple dislocation networks which are amenable to exact analysis. We have determined that a multi-scale model, coupling dislocation dynamics analysis with continuum plasticity, and the right external boundary-conditions are necessary to adequately capture the behavior of finite dislocation boundaries in a finite simulation cell. These numerical analyses were substantiated by simulating infinite and idealized boundaries for which analytical solutions are known (*e.g.*, Li, 1962; Hirth and Lothe 1968; Saada 1979; Saada and Bouchaud, 1993; Lubarda *et al.*, 1993) and for perfect tilt boundaries in a finite slab (Surh and Wolfer, 2001). Boundary and internal stress fields have also been investigated experimentally by x-rays (e.g., Mughrabi *et al.*, 1986).

Long multi-scale simulations runs are under progress to analyze the dynamic behavior of a single dislocation-boundary with hybrid “external-boundary”-conditions for the simulation-cell. In the hybrid boundary-conditions, the boundary-conditions for the four faces of the simulation cell perpendicular to the dislocation boundary are symmetric, while the other two are rigid. Note that periodic boundary-conditions cannot be applied due to the lack of appropriate

symmetry in the types of finite boundaries and dislocation geometries we examine. The dislocation boundary configurations are based loosely on TEM observations and contain three independent Burgers vectors.

Further work will focus on analyzing the interaction of two dislocation boundaries for the two cases when both dislocation-boundaries have similar dislocation contents with the net Burgers vector of the same sign or with the opposite sign (Fig. 26), and when the adjacent boundaries have completely different dislocation contents. Stability analysis for one and two boundaries will also be done in the presence of a Frank Read source. As before we will perform dislocation simulations using as input preconstructed dislocation configurations and boundaries. These configurations include: Three dimensional dislocation clusters, planar dislocation boundaries and arrays of nonplanar walls e.g., cell structures, that enclose nearly dislocation free volumes. Most of these configurations will be based on our transmission electron microscopy observations. Parallel to this computational effort, we will work on the development of a statistical continuum theory that can be used to model the deformation process at mesoscale. The theory will be based on a gradient-plasticity framework recently developed by Zbib and co-workers (Morita *et al*.., 2001; Shizawa *et al*., 2001). The new element is to incorporate statistical data into this framework, i.e. the spatial distributions and evolutions of dislocation boundaries, and the resulting scaling laws.

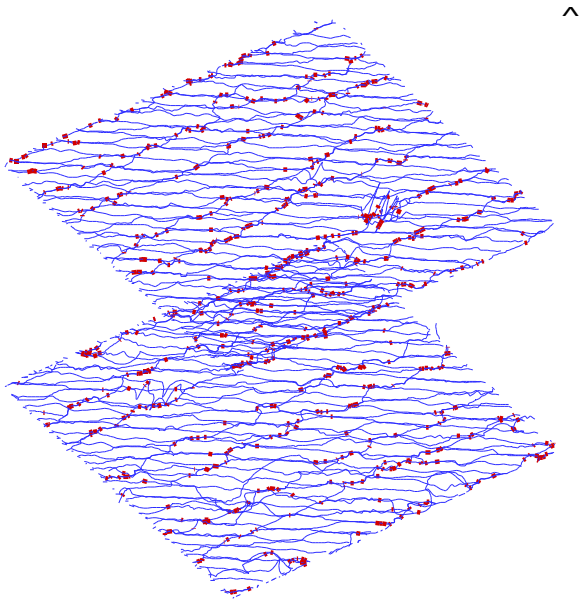

Fig. 26 Two dislocation boundaries with a tilt twist character from the multiscale simulation showing in a relaxed configuration

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